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THE FATE OF TRACE METALS IN A ROTARY KILN INCINERATOR WITH A VENTURI/PACKED COLUMN SCRUBBER

Volume I — Technical Results

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16. ABSTRACT

A five week series of pilot-scale incineration tests, using a synthetic waste feed, was performed at EPA's Incineration Research Facility to evaluate the fate of trace metals fed to a rotary kiln incinerator. Eight tests studied the fate of five hazardous constituent and four nonhazardous constituent trace metals as a function of incinerator operating temperatures and feed chlorine content. Three tests evaluated the valance state of chromium in emissions and discharges as a function of feed valance state and feed chlorine content.

Parametric tests confirmed that cadmium, lead and bismuth are relatively volatile, based on normalized discharge distribution data. Barium, copper, strontium, chromium and magnesium are relatively nonvolatile. Apparent scrubber efficiencies generally correlated with observed volatilities; collection efficiency was higher for nonvolatile metals than for volatile metals. Increased feed chlorine content significantly increased the volatility of cadmium, lead and bismuth.

Chromium test results indicated that with no feed chlorine, 95 percent of the measured chromium is discharged in the kiln ash. With chlorine in the feed, this fraction dropped to 85 percent. Kiln ash contained negligible hexavalent chromium [Cr(+6)] for all tests. The fraction of scrubber exit flue gas chromium as Cr(+6) was nominally 15 percent with no feed chlorine, increasing to 50 percent with chlorine-containing feed.

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FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. These laws direct the EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory is responsible for planning, implementing, and managing research, development, and demonstration programs to provide an authoritative, defensible, engineering basis in support of the policies, programs, and regulations of the EPA with respect to drinking water, wastewater, pesticides, toxic substances, solid and hazardous wastes, and Superfund-related activities. This publication is one of the products of that research and provides a vital communication link between the researcher and the user community.

This report describes an extensive series of tests conducted at the EPA's Incineration Research Facility to evaluate the fate of trace metals fed to a rotary kiln incinerator. For further information, please contact the Waste Minimization, Destruction and Disposal Research Division of the Risk Reduction Engineering Laboratory.

E. Timothy Oppelt, Director
Risk Reduction Engineering Laboratory

ABSTRACT

A 5-week series of pilot-scale incineration tests, employing a synthetic waste feed, was performed at the U.S. Environmental Protection Agency's (EPA) Incineration Research Facility (IRF) to evaluate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber. Completed were three tests to evaluate the valence state of chromium in emissions and discharges as a function of valence state in the feed and feed chlorine content, and eight tests to evaluate the fate of five hazardous constituent and four nonhazardous constituent trace metals as a function of incinerator operating temperatures and feed chlorine content.

Chromium test results indicated that when no chlorine was present in the feed, 95 percent of the measured chromium was discharged in the kiln ash, 1 to 2 percent in the scrubber exit flue gas, and 3 percent in the scrubber liquor. With chlorine in the feed, these fractions were 84, 4, and 11 percent, respectively. Kiln ash contained negligible Cr(+6) for all tests. Scrubber exit flue gas Cr(+6) as a fraction of total chromium was nominally 15 percent with no feed chlorine, increasing to about 50 percent with chlorine-containing feed. The scrubber liquor Cr(+6) fraction was 20 to 30 percent with Cr(+3) feed, increasing to about 60 percent with Cr(+6) feed.

Parametric trace metal test results confirm that cadmium, lead, and bismuth are relatively volatile metals, based on normalized discharge distribution data. Less than 32 percent of these metals were discharged in the kiln ash. Barium, copper, strontium, chromium, and magnesium are relatively nonvolatile, with greater than 75 percent of their discharge amounts present in kiln ash. Surprisingly, arsenic was found to be relatively nonvolatile. Apparent scrubber collection efficiencies generally correlated with observed volatility. Volatile metals exhibited collection efficiencies of 36 to 45 percent; nonvolatile metals, with the exception of copper, exhibited collection efficiencies of 49 to 88 percent. Feed chlorine content had a major effect on the observed volatility of cadmium, lead, and bismuth, with volatility increasing with increased feed chlorine content. Afterburner exit flue gas particle size distributions shifted to smaller size when chlorine was added to the feed.

The average mass balance closures around the kiln ash/scrubber discharge ranged from 48 to 96 percent for individual metals. Overall average closure was 71 percent. From past experience, trace metals mass balance closure results for combustion sources are in the 30 to 200 percent range.

This report was submitted in fulfillment of Contract Number 68-03-3267 by Acurex Corporation under the sponsorship of the U.S. Environmental Protection Agency. This report covers work conducted during August and September, 1988.

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SECTION 1

INTRODUCTION

The RCRA hazardous waste incinerator performance standards promulgated by EPA in January 1981 established limits on incinerator particulate and HCl emissions, and on hazardous organic constituent emissions by requiring that at least 99.99 percent destruction and removal efficiency (DRE) be achieved for the principal organic hazardous constituents (POHCs) in wastes fed to an incinerator. Emissions of hazardous constituent trace metals were not regulated by these standards except in a global sense via the particulate standard. Risk assessments to date have suggested that, of the total risk to human health and the environment from properly operated incinerators, hazardous constituent trace metal emissions may represent the largest component.

Despite its importance, the data base on trace metal emissions from incinerators is currently very sparse. Data on the effects of waste composition and incinerator operation on trace metal emissions are particularly lacking. In response to these data needs, an extensive series of tests was conducted at EPA's Incineration Research Facility (IRF), with funding support from the Office of Solid Waste (OSW), to investigate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber for particulate and acid gas control.

A primary objective of the tests was to investigate the fate of five hazardous constituent trace metals fed in a solid waste matrix to a rotary kiln incinerator as a function of incinerator operating temperatures and feed chlorine content. Of interest were metal partitioning, particle size distribution, flue gas phase distribution, and scrubber efficiency for each of the metals. The trace metals investigated were arsenic, barium, cadmium, chromium, and lead.

In another OSW sponsored effort within EPA's Risk Reduction Engineering Laboratory (RREL) to support trace metal emission regulation development, a numerical model is being developed by another EPA contractor to aid in predicting the relative distributions of trace metals in emissions and discharges from incinerators. Thus, a second objective of this test program was to supply data to evaluate the predictive capabilities of this model, and to perhaps guide further model refinement. To support this objective, an additional four nonhazardous constituent trace metals were included in the test feed material. These were bismuth, copper, magnesium, and strontium.

Finally, there has been a continuing interest in establishing which valence state of chromium predominates in emissions and discharges from incinerators treating chromium-containing wastes. Of the two common chromium valence states, [the trivalent (Cr(+3)) and the hexavalent (Cr(+6))], the hexavalent is much more toxic. In the absence of data showing which form is emitted from an incinerator, risk assessments have generally assumed that the entire amount emitted is in the hexavalent form. This assumption has resulted in specifying conservative emission limits in the regulatory development process. Thus, a third objective of this test program was to develop data on the valence state distribution of chromium in emissions and discharges as a function of feed composition and chromium valence state fed.

The test program to address the above objectives was comprised of both a series of eight parametric tests in which the test waste feed contained nine trace metals, and a series of three tests in which the test waste feed contained either Cr(+3) or Cr(+6).

This report summarizes the results of the test program. Section 2 describes the rotary kiln incineration system at the IRF in which the tests were performed, outlines the test waste feed characteristics, and summarizes the incinerator operating conditions for each of the tests. Section 3 discusses the emissions and discharge stream sampling and analysis protocols. Section 4 presents test program results. Test conclusions are summarized in Section 5. Quality assurance and quality control aspects of the test program are discussed in Section 6.

SECTION 2

FACILITY DESCRIPTION AND TEST CONDITIONS

The pilot-scale rotary kiln incinerator system (RKS) at the IRF in Jefferson, Arkansas, was used in this test program. A description of the system is presented in Section 2.1, followed by a description of the synthetic waste mixture in Section 2.2. The test matrices and operating conditions are described in Section 2.3.

2.1 ROTARY KILN INCINERATOR SYSTEM DESCRIPTION

As indicated in Figure 1 and Table 1, the RKS consists of a primary combustion chamber, a transition section, and a fired afterburner chamber. The primary air pollution control system (APCS) consists of a quench section, a venturi scrubber, and a packed-column scrubber. Quench section installation was completed immediately prior to the initiation of this test series to allow the RKS to more closely reflect typical field installations of rotary kiln/venturi scrubber designs. The quench section modification is discussed further in Section 2.1.2. Downstream of the primary APCS a backup APCS, consisting of a carbon-bed adsorber and a high-efficiency particulate (HEPA) filter, is in place. The backup system is designed to ensure that organic compound emissions and particulate emissions to the atmosphere are negligible during less than optimal test conditions. The main components of the RKS and its APCS are discussed in more detail in the following subsections.

2.1.1 Incinerator Characteristics

The rotary kiln combustion chamber has an inside diameter of 0.95 m (37.5 in) and is 2.1 m (7 ft) long. The chamber consists of 13 cm (5 in) of refractory encased in a 6.3 mm (0.25 in) thick steel shell. The chamber volume, including the transition sections, is 1.74 m^3 (61.4 ft^3). Four steel rollers support the kiln barrel. A variable-speed DC motor coupled with a reducing gear transmission tumbles the rotary kiln. Typical rotation speeds range from 0.2 to 1.5 rpm.

The afterburner chamber has a 0.91 m (3 ft) inside diameter and is 2.74 m (9 ft) long. The afterburner chamber wall consists of a 15 cm (6 in) layer of refractory encased in a 6.3 mm (0.25 in) thick carbon steel shell. The volume of the afterburner chamber is 1.80 m^3 (63.6 ft^3).

An American Combustion, Inc. burner system was used in the RKS for these tests. The system consisted of propane-fired burners in the kiln and afterburner sections, with computer-based gas metering and process control systems designed to control burner flows of propane and air. A separate system was used to control scrubber system operation. Both control systems were interfaced with a personal computer for data acquisition at 20-second intervals.

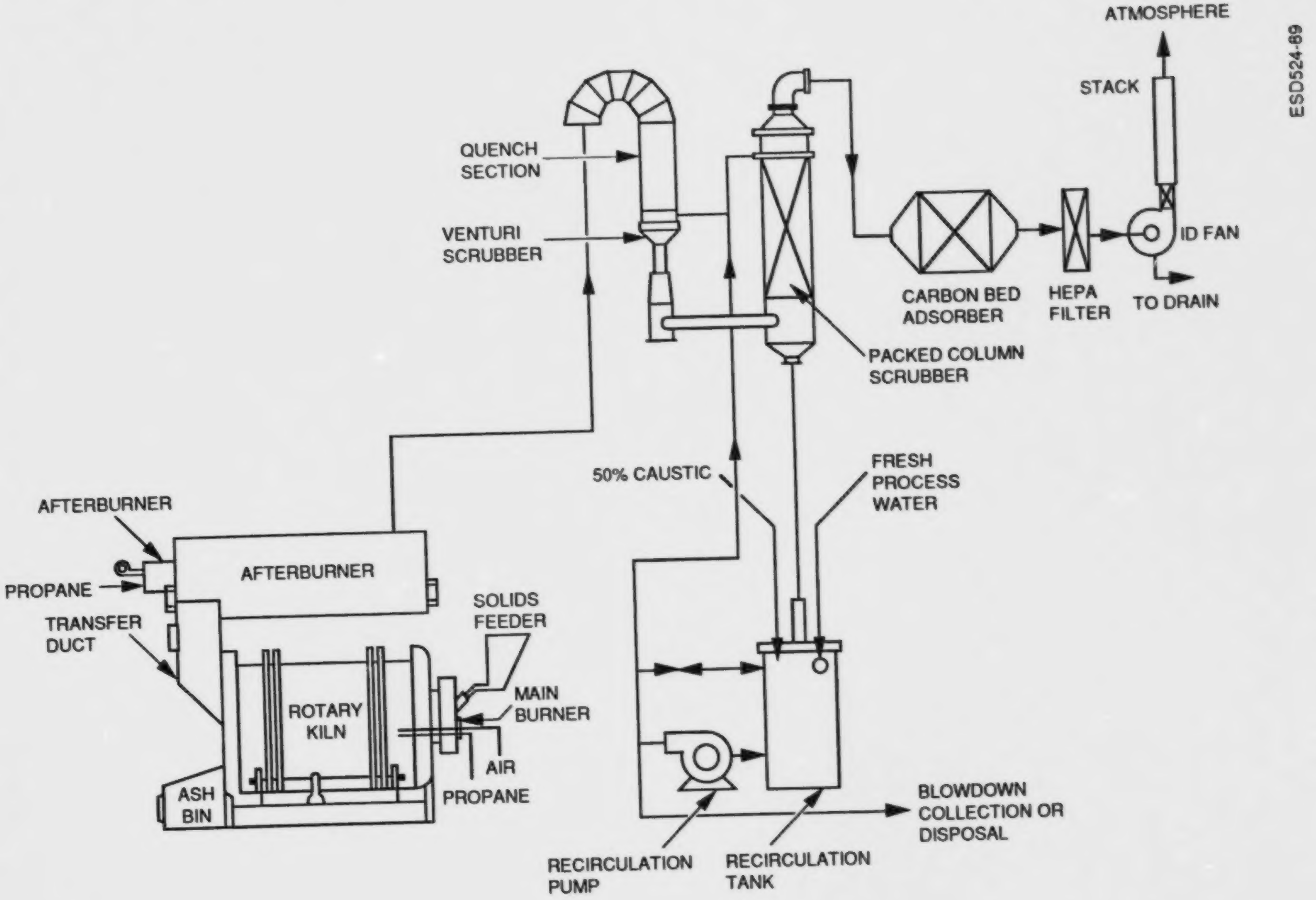

ATMOSPHERE
STACK
ESD524-89
QUENCH SECTION
VENTURI SCRUBBER
ID FAN
CARBON BED ADSORBER
HEPA FILTER
TO DRAIN
PACKED COLUMN SCRUBBER
50% CAUSTIC
FRESH PROCESS WATER
AFTERBURNER
AFTERBURNER
PROPANE
TRANSFER DUCT
SOLIDS FEEDER
MAIN BURNER
ROTARY KILN
AIR
PROPANE
ASH BIN
BLOWDOWN COLLECTION OR DISPOSAL
RECIRCULATION PUMP
RECIRCULATION TANK

Figure 1. Schematic of the IRF RKS.

TABLE 1. DESIGN CHARACTERISTICS OF THE IRF ROTARY KILN SYSTEM

Characteristics of the Kiln Main Chamber

Length, outside	2.61 m (8 ft - 7 in)
Diameter, outside	1.22 m (4 ft)
Length, inside	2.13 m (7 ft)
Diameter, inside	0.95 m (3 ft - 1-1/2 in)
Chamber volume	1.74 m^3 (61.36 ft^3)
Construction	0.63 cm (0.25 in) thick cold rolled steel
Refractory	12.7 cm (5 in) thick high alumina castable refractory, variable depth to produce a frustroconical effect for moving solids
Rotation	Clockwise or counterclockwise 0.2 to 1.5 rpm
Solids retention time	1 hr (at 0.2 rpm)
Burner	American Combustion Burner, rated at 880 kW (3.0 MMBtu/hr) with dynamic O_2 enhancement capability
Primary fuel	Propane
Feed system	
Liquids	Positive displacement pump via water-cooled lance
Sludges	Moyno pump via front face, water-cooled lance
Solids	Metered twin-auger screw feeder or fiber pack ram feeder
Temperature (max)	900°C (1,650°C)

Characteristics of the Afterburner Chamber

Length, outside	3.05 m (10 ft)
Diameter, outside	1.22 m (4 ft)
Length, inside	2.74 m (9 ft)
Diameter, inside	0.91 m (3 ft)
Chamber volume	1.80 m^3 (63.6 ft^3)
Construction	0.63 cm (0.25 in) thick cold rolled steel
Refractory	15.24 cm (6 in) thick high alumina castable refractory
Gas Residence Time	1.2 to 2.5 seconds depending on temperature and excess air
Burner	American Combustion Burner rated at 440 kW (1.5 MMBtu/hr) with dynamic O_2 enhancement capability
Primary fuel	Propane
Temperature (max)	1,200°C (2,200°F)

Characteristics of the Air Pollution Control System

System capacity	
Inlet gas flow	107 m^3/min (3,773 acfm) at 1,200°C (2,200°F) and 101 kPa (14.7 psia)
Pressure drop	
Venturi scrubber	7.5 kPa (30 in WC)
Packed column	1.0 kPa (4 in WC)
Liquid flow	
Venturi scrubber	77.2 L/min (20.4 gpm) at 69 kPa (10 psig)
Packed column	116 L/min (30.6 gpm) at 69 kPa (10 psig)
pH control	Feed back control by NaOH solution addition

2.1.2 Air Pollution Control System

As previously mentioned, combustion gas exiting the afterburner flowed through a primary APCS consisting of a quench section, a venturi scrubber, and a packed column scrubber, then through a secondary APCS consisting of a carbon bed adsorber followed by a HEPA filter. As originally installed, the afterburner exit flue gas was not quenched prior to passing through the venturi scrubber; the venturi scrubber itself was used to quench the flue gas. This is atypical of a conventional rotary kiln/venturi scrubber installation. In addition, since flue gas quenching occurred in the venturi scrubber, its particulate removal efficiency was adversely affected. To rectify this situation, the RKS APCS was modified prior to this test program by installing a quench section upstream of the venturi scrubber. With the quench section installed, the RKS more nearly reflects traditional rotary kiln/venturi scrubber installation designs; and particulate removal characteristics of the venturi scrubber more nearly reflect the capabilities of the technology.

The quench section reduces the temperature of the combustion gas to approximately 77°C (170°F). The cooled flue gas then enters the venturi scrubber which has an automatically adjustable throat. The scrubber is designed to operate at 7.5 kPa (30 in WC) differential pressure, with a maximum liquid flowrate of 77 L/min (20.4 gpm). The scrubber liquor enters at the top of the scrubber and contacts the gas to remove acid gases and entrained particulate.

Downstream of the venturi scrubber, the flue gas enters the packed-column scrubber where additional scrubbing occurs. The scrubber column is packed with 5.1 cm (2 in) diameter polypropylene ballast saddles at a depth of 2.1 m (82 in). It is designed to operate at 1.0 kPa (4 in WC) differential pressure, with a maximum liquid flowrate of 116 L/min (30.6 gpm). Ambient air is also drawn in at the packed-column liquor discharge port. Past experience indicates that induced ambient air comprises approximately 50 percent of the flue gas downstream of the scrubbers.

One liquor recirculation system supplies both the venturi and packed-column scrubbers. The liquor is a dilute NaOH solution and is monitored continuously by a pH sensor. An integral pH controller automatically meters the amount of NaOH needed to maintain the required pH for proper HCl removal.

At the exit of the packed-column scrubber, a demister removes most of the suspended liquid droplets. In a typical commercial incinerator system, the flue gas would be vented to the atmosphere at this point. However, a backup APCS is in place at the IRF. The flue gas passes through a bed of activated carbon designed to adsorb the remaining vapor phase organic compounds. Typically, the carbon bed operates at 77°C (170°F). Because the flue gas is saturated with moisture and is cooled as it flows through the ducts, condensate is continuously formed. The condensate accumulates in the carbon bed and drains through a bottom tap into the blowdown storage tanks.

A set of HEPA filters designed to remove remaining suspended particulate from the flue gas is located downstream of the carbon adsorption bed. An induced draft fan draws and vents the treated effluent gas to the atmosphere.

2.2 SYNTHETIC TEST MIXTURE

The synthetic waste fired during the test program was composed of a mixture of organic liquids added to a clay absorbent material. Trace metals were incorporated by spiking aqueous mixtures of the metals of interest onto the organic liquid-containing solid material. This synthetic waste was fed to the rotary kiln via a screw feeder.

The organic-liquid base (which supplied the heat content and POHC concentrations to the test mixture) consisted of toluene, with varying amounts of tetrachloroethylene and chlorobenzene added to give a range of synthetic waste chlorine contents. Synthetic waste chlorine content was varied from zero (no chlorinated POHCs added) to nominally 8 percent by weight. The synthetic waste was prepared by mixing the organic liquid with the clay absorbent material to yield a mixture containing about 30 percent (weight) organic liquid. After mixing, the test mixture was poured into 55-gallon drums, each holding approximately 140 kg (300 lbs).

The trace metals of interest were prepared in aqueous solutions. These solutions were then metered into the clay/organic liquid matrix at the screw feeder introducing the waste into the kiln. For the parametric series of tests aimed at evaluating the distribution of the five hazardous constituent and four nonhazardous constituent metals in emissions and discharges, the aqueous trace metal feed solution was metered at a rate which would produce the final nominal synthetic waste feed concentrations noted in Table 2 (assuming no background metal concentration in the clay matrix). For the chromium valence state tests, the aqueous chromium feed solution was metered at a rate which would produce a synthetic waste feed concentration of about 50 ppm Cr(+3 or +6) (again assuming zero background clay chromium content).

2.3 TEST CONDITIONS

As noted in Section 1, the test program consisted of two test series: a three-test chromium valence state series, and an eight-test parametric trace metals series. For all tests, an auger was used to feed the clay/organic mixture at a nominal rate of 63 kg/hr (140 lbs/hr). The metals were premixed in an aqueous solution, then added to the clay/organic liquid feed just before introducing the mixture into the kiln. A gear pump continuously injected the trace metal aqueous solution spike into the feed at the auger to produce the desired spike concentrations. The nominal flowrate through the gear pump was 0.5 to 1 L/hr.

The test variables selected for the three chromium valence state tests were feed chlorine content and the chromium valence state in the waste feed. Two tests were performed with chromium fed in the trivalent state. One of these had a waste feed chlorine content of zero; the other had a waste feed chlorine content of nominally 8 percent. The third test was performed with the aqueous spike chromium fed in the hexavalent state with no chlorine in the feed. All three tests were performed under the same nominal incinerator operating conditions: kiln temperature of 871°C (1,600°F), kiln exit flue gas O_2 of 11.5 percent, afterburner temperature of 1,093°C (2,000°F), and afterburner exit flue gas O_2 of 7.5 percent. These conditions are considered typical of industrial hazardous waste incinerator operation.

The test variables chosen for the parametric test series were feed chlorine content, kiln temperature, and afterburner temperature. A factorial experimental matrix for three variables over three levels was chosen, giving eight target test conditions, as shown in Table 3. All tests were designed for the same nominal excess air levels. These were kiln exit flue gas O_2 of 11.5 percent and afterburner exit flue gas O_2 of 7.5 percent.

The actual kiln and afterburner operating conditions achieved for each test are summarized in Tables 4 and 5, respectively. Table 6 provides a similar summary of the air pollution control system operation for each test. In the tables, the chromium valence state tests are designated as Tests 1, 2, and 3; the parametric tests are designated as Tests 4 through 11.

TABLE 2. TARGET TRACE METAL FEED CONCENTRATIONS FOR THE PARAMETRIC TESTS

Metal	Synthetic solid hazardous waste concentration (ppm)
Hazardous constituent trace metals	
Arsenic	50
Barium	40
Cadmium	10
Chromium	50
Lead	50
Other trace metals	
Bismuth	140
Copper	500
Magnesium	80
Strontium	250

TABLE 3. TARGET TEST CONDITIONS

Test	Feed Cl content (%)	Kiln exit temperature, °C (°F)	Afterburner exit temperature, °C (°F)
Chromium Valence State Tests[a]			
1	0	871 (1600)	1093 (2000)
2	0	871 (1600)	1093 (2000)
3	8	871 (1600)	1093 (2000)
Parametric Trace Metal Tests			
4	0	871 (1600)	1093 (2000)
5	4	816 (1500)	1093 (2000)
6	4	927 (1700)	1093 (2000)
7	4	871 (1600)	1093 (2000)
8	4	871 (1600)	1204 (2200)
9	4	871 (1600)	982 (1800)
10[b]	4	871 (1600)	1093 (2000)
11	8	871 (1600)	1093 (2000)

[a] Test 1 performed with hexavalent chromium; tests 2 and 3 performed with trivalent chromium.
[b] Test point 10 is a duplicate of test point 7.

TABLE 4. KILN OPERATING CONDITIONS

Parameter	Test 1 (9-29-88)	Test 2 (9-28-88)	Test 3 (9-26-88)	Test 4 (9-14-88)	Test 5 (8-25-88)	Test 6 (9-16-88)
Propane feedrate						
range, scm/hr	5.1 to 6.8	5.7 to 7.1	7.4 to 9.1	4.5 to 6.2	1.1 to 6.5	7.4 to 8.8
(scfh)	(180 to 240)	(200 to 250)	(260 to 320)	(160 to 220)	(40 to 230)	(260 to 310)
average, scm/hr	6.2	6.4	7.9	5.0	5.4	7.8
(scfh)	(217)	(224)	(280)	(176)	(190)	(275)
Combustion air flowrate						
range, scm/hr	188 to 235	201 to 251	266 to 321	156 to 217	9 to 190	257 to 304
(scfh)	(6630 to 8310)	(7110 to 8850)	(9400 to 11,330)	(5520 to 7650)	(320 to 6700)	(9090 to 10,720)
average, scm/hr	219	226	283	173	157	274
(scfh)	(7739)	(7994)	(9996)	(6095)	(5542)	(9661)
Exit temperature						
range, °C	856 to 893	839 to 893	854 to 886	851 to 892	793 to 843	709 to 946
(°F)	(1572 to 1640)	(1542 to 1639)	(1569 to 1626)	(1564 to 1638)	(1459 to 1550)	(1669 to 1735)
average, °C	873	872	871	874	825	928
(°F)	(1603)	(1602)	(1600)	(1606)	(1517)	(1702)
Pressure						
range, Pa	-3 to -25	-5 to -12	-3 to -45	-5 to -16	-5 to -12	0 to -8
(in WC)	(-0.01 to -0.18)	(-0.02 to -0.05)	(-0.01 to -0.18)	(-0.02 to -0.07)	(-0.02 to -0.05)	(0.00 to -0.03)
average, Pa	-10	-6	-7	-9	-8	-6
(in WC)	(-0.03)	(-0.02)	(-0.03)	(-0.04)	(-0.03)	(-0.02)
Exit O_2						
range, %	10.7 to 11.8	10.9 to 11.8	11.1 to 13.2	11.8 to 12.8	11.0 to 13.6	9.0 to 11.0
average, %	11.2	11.3	11.5	12.2	11.9	10.6
Clay/organic mixture feedrate						
total, kg/hr	62.6	62.0	61.3	59.4	63.5	62.6
(lb/hr)	138.0	136.7	135.1	131.0	140.0	138.0
organic fraction,						
kg/hr	17.5	17.5	18.4	13.8	15.0	17.2
(lb/hr)	38.5	38.5	40.5	30.4	33.0	38.0

(continued)

TABLE 4. (concluded)

Parameter	Test 7 (8-30-88)	Test 8 (9-7-88)	Test 9 (9-9-88)	Test 10 (9-20-88)	Test 11 (9-22-88)
Propane feedrate					
range, scm/hr	6.2 to 7.9	7.1 to 10.2	5.4 to 6.8	4.8 to 7.1	7.1 to 9.3
(scfh)	(220 to 280)	(250 to 360)	(190 to 240)	(170 to 250)	(250 to 330)
average, scm/hr	6.9	9.2	6.1	6.4	7.5
(scfh)	(244)	(325)	(216)	(225)	(263)
Combustion air flowrate					
range, scm/hr	223 to 277	203 to 269	176 to 243	176 to 257	260 to 330
(scfh)	(7880 to 9780)	(7180 to 9500)	(6230 to 8590)	(6230 to 9070)	(9170 to 11,670)
average, scm/hr	246	245	213	233	271
(scfh)	(8685)	(8643)	(7537)	(8226)	(9570)
Exit temperature					
range, °C	869 to 886	832 to 916	836 to 891	841 to 894	852 to 885
(°F)	(1597 to 1626)	(1530 to 1680)	(1536 to 1636)	(1546 to 1642)	(1565 to 1625)
average, °C	878	871	875	873	870
(°F)	(1612)	(1599)	(1607)	(1603)	(1599)
Pressure					
range, Pa	-5 to -11	-5 to -8	-8 to -20	-3 to -5	0 to -45
(in WC)	(-0.02 to -0.04)	(-0.02 to -0.03)	(-0.03 to -0.08)	(-0.01 to -0.02)	(0.00 to -0.18)
average, Pa	-8	-6	-10	-5	-10
(in WC)	-0.03	(-0.02)	(-0.04)	(-0.02)	(-0.04)
Exit O_2					
range, %	11.7 to 13.7	12.8 to 14.5	12.1 to 13.0	10.6 to 12.7	10.8 to 11.7
average, %	12.4	13.6	12.7	11.4	11.4
Clay/organic mixture feedrate					
total, kg/hr	63.4	63.4	62.5	62.8	63.5
(lb/hr)	139.8	139.8	137.8	138.4	140.0
organic fraction, kg/hr	16.8	15.0	14.1	18.7	18.1
(lb/hr)	37.0	33.0	31.0	41.3	40.0

TABLE 5. AFTERBURNER OPERATING CONDITIONS

Parameter	Test 1 (9-29-88)	Test 2 (9-28-88)	Test 3 (9-26-88)	Test 4 (9-14-88)	Test 5 (8-25-88)	Test 6 (9-16-88)
Propane feedrate						
range, scm/hr	17.0 to 17.8	16.7 to 17.6	17.0 to 17.8	16.4 to 17.3	17.8	16.4 to 17.3
(scfh)	(600 to 630)	(590 to 620)	(600 to 630)	(580 to 610)	(630)	(580 to 610)
average, scm/hr	17.3	17.0	17.3	16.7	17.8	17.1
(scfh)	(609)	(600)	(611)	(591)	(630)	(604)
Combustion air flowrate						
range, scm/hr	534 to 540	510 to 526	491 to 512	540	540	540
(scfh)	(18,860 to 19,060)	(18,020 to 18,570)	(17,330 to 18,070)	(19,060)	(19,060)	(19,060)
average, scm/hr	540	521	497	540	540	540
(scfh)	(19,059)	(18,392)	(17,567)	(19,060)	(19,060)	(19,060)
Exit temperature						
range, °C	1090 to 1103	1091 to 1103	1091 to 1102	1083 to 1100	1052 to 1082	1084 to 1112
(°F)	(1994 to 2018)	(1996 to 2017)	(1996 to 2016)	(1982 to 2012)	(1925 to 1979)	(1984 to 2033)
average, °C	1094	1095	1096	1093	1071	1092
(°F)	(2002)	(2003)	(2005)	(1999)	(1959)	(1998)
Pressure						
range, Pa	-30 to -95	-30 to -40	-15 to -30	-20 to -30	-20 to -50	-25 to -32
(in WC)	(-0.12 to -0.38)	(-0.12 to -0.16)	(-0.06 to -0.10)	(-0.08 to -0.12)	(-0.08 to -0.20)	(0.10 to -0.13)
average, Pa	-42	-32	-27	-25	-42	-30
(in WC)	(-0.15)	(-0.12)	(-0.11)	(-0.10)	(-0.17)	(-0.12)
Exit O_2						
range, %	6.6 to 7.9	6.4 to 8.0	6.3 to 7.7	6.1 to 8.0	6.0 to 10.2	5.7 to 7.8
average, %	7.3	7.4	7.2	7.1	8.4	7.1
Exit CO_2						
range, %	8.9 to 10.1	8.9 to 10.2	9.0 to 10.2	8.6 to 10.2	6.7 to 10.0	8.7 to 10.4
average, %	9.5	9.4	9.5	9.5	8.2	9.4

(continued)

TABLE 5. (concluded)

Parameter	Test 7 (8-30-88)	Test 8 (9-7-88)	Test 9 (9-9-88)	Test 10 (9-20-88)	Test 11 (9-22-88)
Propane feedrate					
range, scm/hr	17.8	17.6 to 17.8	13.0 to 13.3	17.3 to 17.8	17.3 to 17.8
(scfh)	(620 to 630)	(620 to 630)	(460 to 470)	(610 to 630)	(610 to 630)
average, scm/hr	17.8	17.8	13.2	17.5	17.5
(scfh)	(630)	(630)	(466)	(619)	(619)
Combustion air flowrate					
range, scm/hr	540	445 to 540	539 to 540	540	540
(scfh)	(19,060)	(15,730 to 19,060)	(19,050 to 19,060)	(19,060)	(19,060)
average, scm/hr	540	451	540	540	540
(scfh)	(19,060)	(15,912)	(19,060)	(19,060)	(19,060)
Exit temperature					
range, °C	1079 to 1095	1168 to 1213	967 to 991	1081 to 1103	1084 to 1105
(°F)	(1974 to 2003)	(2135 to 2216)	(1791 to 1816)	(1981 to 2014)	(1984 to 2021)
average, °C	1088	1196	983	1094	1092
(°F)	(1991)	(2184)	(1803)	(2000)	(1998)
Pressure					
range, Pa	0 to -55	-30 to -37	-15 to -37	-25	-25 to -95
(in WC)	(0.00 to -0.22)	(-0.12 to -0.15)	(-0.06 to -0.15)	(-0.12)	(-0.10 to -0.38)
average, Pa	-52	-30	-22	-25	-35
(in WC)	(-0.21)	(-0.12)	(-0.09)	(-0.10)	(-0.14)
Exit O_2					
range, %	7.1 to 8.7	3.7 to 7.5	7.7 to 9.3	6.5 to 8.5	6.3 to 8.1
average, %	7.9	5.9	8.7	7.6	7.5
Exit CO_2					
range, %	7.6 to 9.5	8.9 to 11.2	7.7 to 9.0	8.5 to 10.0	4.8 to 6.8
average, %	8.8	10.3	8.3	9.2	6.1

TABLE 6. AIR POLLUTION CONTROL SYSTEM OPERATING CONDITIONS

Parameter	Test 1 (9-29-88)	Test 2 (9-28-88)	Test 3 (9-26-88)	Test 4 (9-14-88)	Test 5 (8-25-88)	Test 6 (9-16-88)
Quench chamber scrubber liquor flowrate						
range, L/min	68	53 to 68	53 to 68	53 to 72	45 to 49	45 to 68
(gpm)	(18.0)	(14.0 to 18.0)	(14.0 to 18.0)	(14.0 to 19.0)	(12.0 to 13.0)	(14.0 to 18.0)
average, L/min	68	66	67	54	45	67
(gpm)	(18.0)	(17.5)	(17.8)	(14.2)	(12.0)	(17.6)
Venturi scrubber liquor flowrate						
range, L/min	76	76 to 79	45 to 87	64 to 68	64 to 68	64
(gpm)	(20.0)	(20.0 to 21.0)	(12.0 to 23.0)	(17.0 to 18.0)	(17.0 to 18.0)	(17.0)
average, L/min	76	76	73	66	68	64
(gpm)	(20.0)	(20.0)	(19.2)	(17.5)	(17.9)	(17.0)
Venturi scrubber pressure drop						
range, kPa	6.0 to 6.2	6.0 to 6.7	4.5 to 6.2	5.7 to 7.0	4.7 to 5.7	5.5 to 6.2
(in WC)	(24 to 25)	(24 to 27)	(18 to 25)	(23 to 28)	(19 to 23)	(22 to 25)
average, kPa	6.0	6.1	5.5	6.0	5.3	5.7
(in WC)	(24.1)	(24.6)	(22.0)	(24.2)	(21.2)	(23.0)
Packed column scrubber liquor flowrate						
range, L/min	106	106 to 110	68 to 132	102 to 106	114 to 121	95
(gpm)	(28.0)	(28.0 to 29.0)	(18.0 to 35.0)	(27.0 to 28.0)	(30.0 to 32.0)	(25.0)
average, L/min	106	108	106	104	115	95
(gpm)	(28.0)	(28.5)	(27.9)	(27.6)	(30.4)	(25.0)
Packed column pressure drop						
range, kPa	3.2 to 3.5	2.7 to 3.2	3.0 to 3.7	2.0 to 2.5	0.25 to 1.5	3.0 to 3.7
(in WC)	(13 to 14)	(11 to 13)	(12 to 15)	(8 to 10)	(1 to 6)	(12 to 15)
average, kPa	3.2	3.1	3.6	2.2	0.8	3.4
(in WC)	(13.0)	(12.4)	(14.4)	(8.9)	(3.1)	(13.8)
Scrubber liquor pH						
range	6.5 to 7.1	6.5 to 7.2	6.0 to 9.6	7.0 to 7.3	3.3 to 8.8	6.4 to 8.2
average	7.0	7.0	7.6	7.2	6.8	7.1

(continued)

TABLE 6. (continued)

Parameter	Test 1 (9-29-88)	Test 2 (9-28-88)	Test 3 (9-26-88)	Test 4 (9-14-88)	Test 5 (8-25-88)	Test 6 (9-16-88)
Scrubber blowdown flowrate						
range, L/min	0.8 to 3.0	1.7 to 2.6	1.5 to 6.1	0.0 to 3.0	1.9 to 2.6	0.8 to 3.8
(gpm)	(0.2 to 0.8)	(0.5 to 0.7)	(0.4 to 1.6)	(0.0 to 0.8)	(0.5 to 0.7)	(0.2 to 1.0)
average, L/min	1.9	1.9	2.6	1.9	2.3	1.9
(gpm)	(0.5)	(0.5)	(0.7)	(0.5)	(0.6)	(0.5)
Scrubber liquor temperature						
range, (°C)	68 to 76	74 to 75	43 to 76	73 to 75	72 to 75	68 to 75
°F	(155 to 169)	(166 to 167)	(109 to 168)	(164 to 167)	(162 to 167)	(155 to 167)
average, (°C)	74	75	73	74	73	74
°F	(166)	(167)	(163)	(166)	(164)	(166)
ID fan inlet pressure						
range, kPa	-6.0 to -9.0	-8.5 to -8.7	-8.5 to -8.7	-7.5 to -8.5	-5.5 to -5.7	-8.7
(in WC)	(-24 to -36)	(-34 to -35)	(-34 to -37)	(-30 to -34)	(-22 to -23)	(-35)
average, kPa	-8.3	-8.6	-8.6	-7.6	-5.5	-8.7
(in WC)	(-33.5)	(-34.4)	(-34.4)	(-30.6)	(-22.1)	(-35)

(continued)

TABLE 6. (continued)

Parameter	Test 7 (8-30-88)	Test 8 (9-7-88)	Test 9 (9-9-88)	Test 10 (9-20-88)	Test 11 (9-22-88)
Quench chamber scrubber liquor flowrate					
range, L/min	45	57	49 to 53	53 to 68	68
(gpm)	(12.0)	(15.0)	(13.0 to 14.0)	(14.0 to 18.0)	(18.0)
average, L/min	45	57	53	55	68
(gpm)	(12.0)	(15.0)	(14.0)	(14.4)	(18.0)
Venturi scrubber liquor flowrate					
range, L/min	53 to 72	68	68 to 72	76	72 to 76
(gpm)	(14.0 to 19.0)	(18.0)	(18.0 to 19.0)	(20.0)	(19.0 to 20.0)
average, L/min	69	68	69	76	73
(gpm)	(18.1)	(18.0)	(18.3)	(20.0)	(19.3)
Venturi scrubber pressure drop					
range, kPa	2.5 to 7.0	4.7 to 5.2	5.2 to 6.7	5.2 to 6.2	4.0 to 6.2
(in WC)	(10 to 28)	(19 to 21)	(21 to 27)	(21 to 25)	(16 to 25)
average, kPa	5.7	4.9	6.3	5.6	5.1
(in WC)	(22.8)	(19.8)	(25.4)	(22.6)	(20.6)
Packed column scrubber liquor flowrate					
range, L/min	110 to 114	110 to 114	106 to 110	110 to 114	110 to 114
(gpm)	(29 to 30)	(29 to 30)	(28 to 29)	(29 to 30)	(29 to 30)
average, L/min	111	112	108	113	111
(gpm)	(29.3)	(29.6)	(28.4)	(29.8)	(29.3)
Packed column pressure drop					
range, kPa	2.7 to 3.7	3.2 to 3.7	1.2 to 2.0	3.0 to 3.7	1.5 to 3.7
(in WC)	(11 to 15)	(13 to 15)	(5 to 8)	(12 to 15)	(6 to 15)
average, kPa	3.1	3.6	1.6	3.2	3.4
(in WC)	(12.3)	(14.4)	(6.4)	(12.9)	(13.6)
Scrubber liquor pH					
range	6.0 to 8.9	6.2 to 8.1	5.9 to 8.0	5.1 to 8.2	5.0 to 8.5
average	7.2	7.2	6.9	6.8	7.5

(continued)

TABLE 6. (concluded)

Parameter	Test 7 (8-30-88)	Test 8 (9-7-88)	Test 9 (9-9-88)	Test 10 (9-20-88)	Test 11 (9-22-88)
Scrubber blowdown flowrate					
range, L/min	1.5 to 5.7	0.8 to 9.5	0.0 to 1.9	1.5 to 5.7	1.5 to 3.0
(gpm)	(0.4 to 1.5)	(0.2 to 2.5)	(0.0 to 0.5)	(0.4 to 1.5)	(0.4 to 0.8)
average, L/min	2.3	1.9	1.5	2.3	2.3
(gpm)	(0.6)	(0.5)	(0.4)	(0.6)	(0.6)
Scrubber liquor temperature					
range, (°C)	72 to 74	74 to 76	72 to 74	74 to 76	69 to 77
°F	(161 to 166)	(166 to 169)	(162 to 165)	(165 to 168)	(156 to 171)
average, (°C)	73	76	73	75	75
°F	(164)	(168)	(163)	(167)	(167)
ID fan inlet pressure					
range, kPa	-5.2 to -8.7	-7.7 to -9.7	-6.5 to -7.5	-8.0 to -8.5	-5.0 to -9.2
(in WC)	(-21 to -35)	(-31 to -39)	(-26 to -30)	(-32 to -34)	(-32 to -37)
average, kPa	-7.9	-8.1	-7.4	-8.2	-8.1
(in WC)	(-31.7)	(-32.6)	(-29.7)	(-32.8)	(-32.4)

Control room records of the operating parameters recorded at 15-minute intervals are given in Appendix A. Appendix A also contains plots of the operating conditions for the kiln and afterburner recorded at 20-second intervals on the PC data acquisition system.

The ranges and averages presented in Tables 4 and 5 were developed using the computer recorded data obtained during periods of flue gas sampling for all tests except Test 6. During Test 6 a power outage disabled the data acquisition system midway through the test. The remaining test data were taken from the data log book. The kiln exit O_2 monitor also malfunctioned during this test. However, O_2 readings were obtained at 30-minute intervals using an Orsat analyzer; these values were used to develop the values presented in the tables. The APCS data in Table 6 were taken from the control room log. Only flue gas O_2 and CO_2 are given in Tables 4 and 5. Flue gas concentrations of other combustion gas species (e.g. CO) are presented in Section 4.2.

Table 7 summarizes the actual incinerator operating condition ranges and averages achieved for each test (temperatures and flue gas O_2 levels) and compares these to the respective target conditions. Figure 2 presents a graphical summary of the incinerator temperature data from Table 7.

Figure 2 illustrates that average test temperatures achieved were within 9°C (17°F) of target conditions for all tests except one. For this one test the afterburner temperature was 22°C (41°F) lower than the target. The data in Table 7 show that kiln exit flue gas O_2 was generally within 1 percent of the test program target of 11.5 percent, and that afterburner exit flue gas O_2 was generally within about 1 percent of the test program target of 7.5 percent for all tests except one. For this one test, fan capacity limitations resulted in afterburner exit flue gas O_2 being substantially below the target level, while kiln exit flue gas O_2 was high.

Brief flameouts in the kiln and afterburner did occur during Test 3, generally due to loss of signal from the flame sensor. Clay/organic liquid mixture and metal feeds were stopped while the system was restarted, and flue gas sampling had not yet begun. Therefore, this upset is not expected to affect the data.

TABLE 7. ACTUAL VERSUS TARGET OPERATING CONDITIONS FOR THE TRACE METAL TEST SERIES

		Kiln exit					Afterburner exit				
		Temperature, °C (°F)					Temperature, °C (°F)				
			Actual					Actual			
Test	Test date	Target	Minimum	Maximum	Average	Flue gas O_2 (%)	Target	Minimum	Maximum	Average	Flue gas O_2 (%)
Chromium valence state tests											
1	9/29/88	871 (1600)	856 (1572)	893 (1640)	873 (1603)	11.1	1093 (2000)	1090 (1994)	1103 (2018)	1094 (2003)	7.3
2	9/28/88	871 (1600)	839 (1542)	893 (1639)	872 (1602)	11.2	1093 (2000)	1091 (1996)	1103 (2017)	1095 (2003)	7.3
3	9/26/88	871 (1600)	854 (1569)	866 (1626)	871 (1600)	11.5	1093 (2000)	1091 (1996)	1102 (2016)	1096 (2005)	7.2
Parametric tests											
4	9/14/88	871 (1600)	851 (1564)	892 (1638)	874 (1606)	12.2	1093 (2000)	1083 (1982)	1100 (2012)	1093 (1999)	7.1
5	8/25/88	816 (1500)	793 (1459)	843 (1550)	825 (1517)	11.9	1093 (2000)	1052 (1925)	1082 (1979)	1071 (1959)	8.4
6	9/26/88	927 (1700)	909 (1669)	946 (1735)	928 (1701)	10.6[a]	1093 (2000)	1084 (1984)	1112 (2033)	1092 (1998)	7.1
7	8/30/88	871 (1600)	869 (1597)	886 (1626)	878 (1612)	12.4	1093 (2000)	1079 (1974)	1095 (2003)	1088 (1991)	7.9
8	9/07/88	871 (1600)	832 (1530)	916 (1680)	871 (1599)	13.6	1204 (2200)	1168 (2135)	1213 (2216)	1196 (2184)	5.8[b]
9	9/09/88	871 (1600)	836 (1536)	891 (1636)	875 (1607)	12.7	982 (1800)	967 (1791)	991 (1816)	983 (1803)	8.7
10	9/20/88	871 (1600)	841 (1546)	894 (1642)	873 (1603)	11.4	1093 (2000)	1081 (1981)	1103 (2014)	1094 (2000)	7.6
11	9/22/88	871 (1600)	852 (1565)	885 (1625)	870 (1599)	11.4	1093 (2000)	1084 (1984)	1105 (2021)	1092 (1998)	7.4

[a] Monitor not in service. Data obtained using Bacharach Fyrite O_2 analyzer.

[b] Afterburner air feedrate at fan capacity.

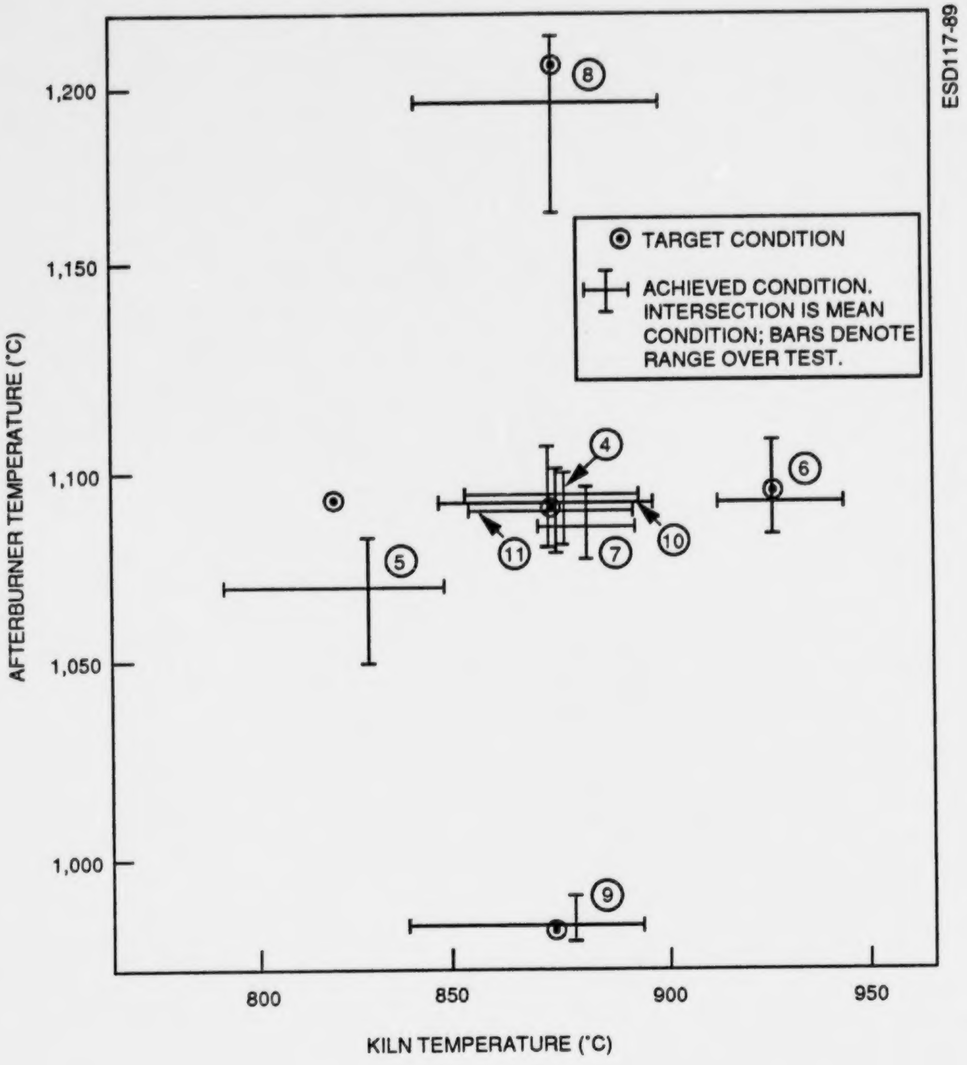


Figure 2. Actual versus target operating temperatures for the parametric trace metal tests.

SECTION 3

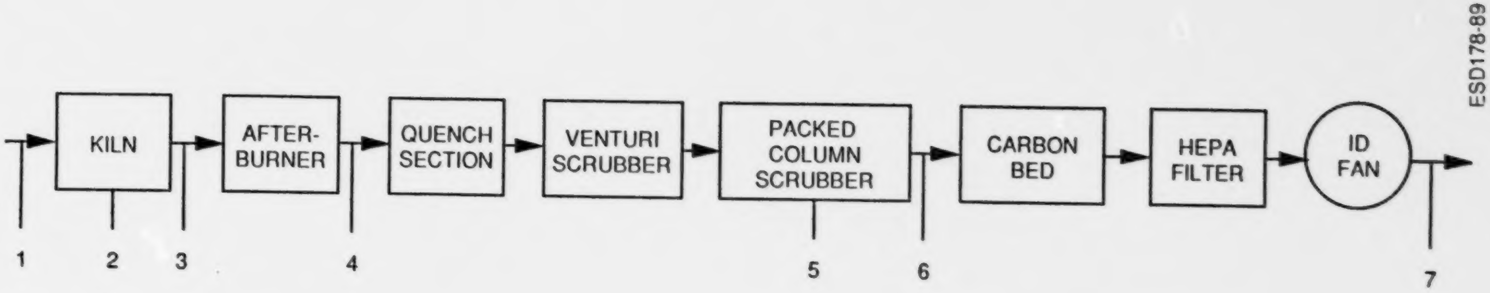
SAMPLING AND ANALYSIS PROCEDURES

Sampling and analysis for both the chromium valence state- and parametric test series were designed to meet research objectives and IRF permit-compliance requirements. Sampling locations were the same for both test series, but specific analysis methods were used for the different trace metals. Figure 3 identifies the sampling point locations. Stack sampling (sampling point 7) was primarily for permit compliance. In general, sampling for each test consisted the following:

- Obtaining a composite sample of the feed materials (clay/organic liquid mixture, and aqueous metal spike solution) and of the ash
- Obtaining several samples of the scrubber blowdown water over time
- Obtaining samples of the flue gas at the afterburner and scrubber exits for particulate and vapor phase metal analysis
- Obtaining samples of the flue gas at the afterburner and scrubber exits for volatile organic hazardous constituent analyses using a volatile organic sampling train (VOST)
- Obtaining continuous monitor sampling of various combinations of flue gas O_2, CO, CO_2, NO_x, and total unburned hydrocarbon (TUHC) at the kiln, afterburner, and scrubber exits and in the stack
- Obtaining samples of the flue gas at the afterburner and scrubber exits, and at the stack for total particulate and HCl

The continuous emission monitors (CEMs) available at the IRF and the locations they monitored for these tests are summarized in Table 8. This monitoring arrangement was employed in all tests. Figure 4 illustrates the generalized flue gas conditioning and flow distribution system at the IRF. Four independent systems, such as those illustrated in Figure 4, are in place so that appropriately conditioned sample gas from four separate sampling locations can be routed to any of the available monitors listed in Table 8. The CEM setup described in Table 8, with appropriate gas conditioning per Figure 4, was employed throughout this test program.

Details of the extractive sampling and analysis procedures are discussed in the following subsections.


KILN
AFTER-BURNER
QUENCH SECTION
VENTURI SCRUBBER
PACKED COLUMN SCRUBBER
CARBON BED
HEPA FILTER
ID FAN
1
2
3
4
5
6
7
ESD178-89

SAMPLE LOCATIONS

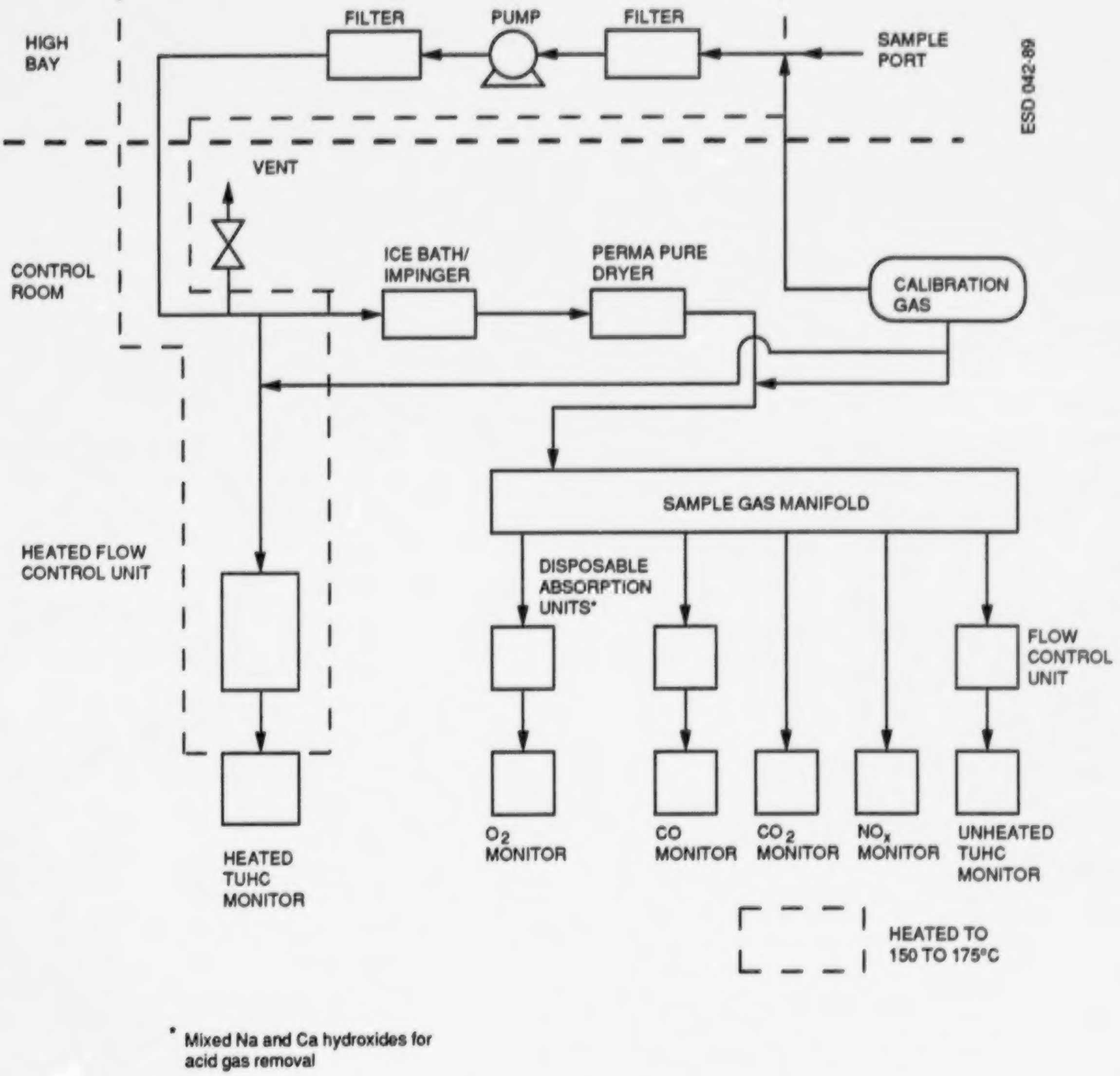
	Feeds and Residuals				Continuous Monitors					Flue Gas					
Sampling Point	Clay Organic Liquid	Metal Spike Solution	Kiln Ash	Scrubber Blowdown	O_2	CO, CO_2	NO_x	Unheated TUHC	Heated TUHC	Method 5 (Tests 1-3) with multiple metals impingers, (particulate and Cr)	Simultaneous Method 5 (Tests 1-3) with no filter and caustic impingers (Cr(+6) and HCl)	Method 17 (Tests 4-11) with multiple metals impingers (particulate, metals and HCl)	Method 5 (Tests 4-11) with multiple metals impingers (particulate, metals, and HCl)	VOST (volatile organics)	Method 5 (particulate and HCl)
1	X	X													
2			X												
3					X										
4					X	X		X		X	X	X		X[a]	
5				X											
6						X	X	X	X	X	X		X	X[a]	
7					X	X			X					X[b]	X

[a] Tests 4 through 11 only
[b] Tests 5, 8, and 11 only

Figure 3. Sampling summary for the chromium valence state and parametric trace metals test series.

TABLE 8. CONTINUOUS EMISSION MONITORS USED FOR THE TESTS

		Monitor			
Location	**Constituent**	**Manufacturer**	**Model**	**Principle**	**Range**
Kiln exit	O_2	Beckman	755	Paramagnetic	0-10 percent 0-25 percent 0-50 percent 0-100 percent
Afterburner exit	O_2	Beckman	755	Paramagnetic	0-10 percent 0-25 percent 0-50 percent 0-100 percent
	CO	Horiba	VIA 500	NDIR	0-50 ppm 0-500 ppm
	CO_2	Horiba	PIR 2000	NDIR	0-20 percent 0-80 percent
	Unheated TUHC	Shimadzu	GC Mini	FID	0-10 ppm to 0-2,000 ppm in multiples of 2
Scrubber exit	CO	Horiba	VIA 500	NDIR	0-50 ppm 0-500 ppm
	CO_2	Horiba	PIR 2000	NDIR	0-20 percent 0-80 percent
	NO_x	Thermo Electron	10AR	Chemilumi nescent	0-75 ppm to 0-10,000 ppm in multiples of 2
	Unheated TUHC	Shimadzu	GC Mini	FID	0-10 ppm to 0-2,000 ppm in multiples of 2
	Heated TUHC	Shimadzu	GC Mini	FID	0-10 ppm to 0-2,000 ppm in multiples of 2
Stack	CO/CO_2	Infrared Industries	7020	NDIR	0-20 percent CO_2 0-2,000 ppm CO
	Heated TUHC	Shimadzu	GC Mini	FID	0-10 ppm to 0-2,000 ppm in multiples of 2
	O_2	Teledyne	326A	Fuel cell	0-5 percent 0-10 percent 0-25 percent



HIGH BAY
FILTER
PUMP
FILTER
SAMPLE PORT
ESD 042-89
VENT
CONTROL ROOM
ICE BATH/ IMPINGER
PERMA PURE DRYER
CALIBRATION GAS
SAMPLE GAS MANIFOLD
HEATED FLOW CONTROL UNIT
DISPOSABLE ABSORPTION UNITS*
FLOW CONTROL UNIT
HEATED TUHC MONITOR
O2 MONITOR
CO MONITOR
CO2 MONITOR
NOx MONITOR
UNHEATED TUHC MONITOR
HEATED TO 150 TO 175°C
* Mixed Na and Ca hydroxides for acid gas removal

Figure 4. Generalized CEM sample gas flow schematic.

3.1 CHROMIUM VALENCE TESTS

Table 9 summarizes the sampling and analysis matrix in addition to the CEM monitoring for the chromium valence state test series.

3.1.1 Sampling Procedures

As indicated in Table 9, the flue gas at two locations, the afterburner exit and the scrubber exit, was simultaneously sampled for particulate, total chromium (Cr), hexavalent chromium Cr(+6), and HCl (Test 3 only), using variations of a standard Method 5 sampling train. Two sampling trains were simultaneously operated at each location. One train at each location sampled for particulate load and total chromium levels. The other train at each location sampled for Cr(+6) and, during Test 3, HCl. Particulate load and HCl were also measured at the stack for all tests, using a standard Method 5 train.

The impingers in the train used for particulate and total chromium capture were specified for multiple metals sampling as noted in Table 10. After sampling, the train contents were collected as specified in Method 5. The probe wash (acetone) and filter were desiccated to constant weight to provide the particulate load measurement. After final weighing, the probe wash was resuspended in acetone for later metal analysis. The aqueous condensate from the first impinger was preserved to a pH of less than 2 with HNO_3.

The impinger contents of the train used for Cr(+6) sampling were as noted in Table 11. This train did not contain a filter since Cr(+6) in flue gas samples is best preserved if it is collected in an alkaline solution as quickly as possible. A NaOH solution was used to wash the sampling probe. This solution was then combined with the solution in impinger 1. Aliquots of the impinger 1, 2, and 3 solutions were taken for HCl measurement for Test 3.

All Method 5 trains used glass-lined sampling probes. The trains at the afterburner and scrubber exits collected at least 2.8 m^3 (100 ft^3) of flue gas over about a 3-hour sampling period. This period began no less than 1/2 hour after the start of the test mixture feed. Test mixture feed continued until sampling was complete. The sampling trains at the stack collected at least 0.9 m^3 (32 ft^3).

Grab sampling of chromium spike solution (50 mL) and scrubber blowdown (100 mL) was performed at the beginning of Method 5 sampling. Additional blowdown grab samples were taken hourly until all Method 5 sampling was complete. The chromium spike solution and each blowdown sample were split into two samples, one for total chromium and one for Cr(+6) analysis. At the conclusion of each test, two composite samples of the kiln ash were collected from the ash pit, one for total chromium and one for Cr(+6) analysis. All spike solution and blowdown samples for total chromium analysis were preserved with HNO_3 to $pH<2$. All blowdown samples for Cr(+6) analysis were preserved with NaOH solution to $pH>12$. Kiln ash samples for Cr(+6) analysis were preserved with 3 percent Na_2CO_3, 2 percent NaOH solution (the solution used for Cr(+6) digestion in Method 3060, Reference 2).

Composite feed samples from each drum were collected by trier sampling at three locations in the drum cross-section. Each of these composites was ultimately analyzed for toluene, tetrachloroethylene, chlorobenzene, and ultimate composition. Samples for volatile organic analysis were collected from each drum's composite sample when the contents of that drum were first fed into the kiln. These samples were sealed in 40 mL VOA vials at that time. An aliquot of each drum's composite sample was also submitted for ultimate analysis and for total chromium analysis.

TABLE 9. SAMPLING AND ANALYSIS MATRIX FOR THE CHROMIUM VALENCE STATE TESTS

Sample	Location	Sampling procedure	Analysis: Parameter	Analysis: Method	Frequency
Aqueous chromium spike solution	Kiln inlet	Composite	Total Cr	Digestion by Method 3010 analysis by Method 7191[a]	1/test
			Cr(+6)	Digestion by Method 3060[b] analysis by Method 7197[a]	1/test
Feed clay	Waste preparation	Composite	Cr(+6)	Digestion by Method 3060[b] analysis by Method 7197[a]	1 composite
Clay/organic liquid mixture	Preparation storage drum	Trier	C_7H_8, C_2Cl_4, C_6H_5Cl	Purge and trap by Method 5030,[a] GC/FID analysis	1/test
			Ultimate analysis (C, H, O, Cl)	A003[c]	1/test
			Total Cr	Parr bomb ashing[d], Digestion by Method 3050[a], analysis by Method 6010[a]	1/test
Kiln ash	Kiln ash pit	Composite	Total Cr	Digestion by Method 3050 analysis by Method 7191[a]	1/test
			Cr(+6)	Digestion by Method 3060[b], analysis by Method 7197[a]	4/test
Scrubber blow-down water	Blowdown discharge	Grab (tap)	Total Cr	Digestion by Method 3010 analysis by Method 7191[a]	4/test
			Cr(+6)	Digestion by Method 3060[b], analysis by 7197[a]	4/test
Flue gas	Afterburner and scrubber exits	Method 5[e]	Particulate load	Method 5[e]	1/test
			Total Cr	Digestion by Method 3010 (impingers) or Method 3050 (particulate), analysis by Method 7191[a]	1/test
		Method 5[e]	HCl	Analysis of impinger solution for Cl by specific ion electrode	Test 3 only
			Cr(+6)	Digestion by Method 3060[b], analysis by Method 7197[a]	1/test
Stack gas	Stack downstream of carbon bed/ HEPA filter	Method 5[e]	Particulate load	Method 5[e]	1/test
			HCl	Analysis of impinger solution for Cl by specific ion electrode	Test 3 only

[a] (1)
[b] (2)
[c] (3)
[d] (4)
[e] (5)

TABLE 10. MULTIPLE METALS TRAIN IMPINGER SYSTEM REAGENTS FOR THE CHROMIUM VALENCE STATE TESTS

Impinger Number	Reagent	Quantity
1	Empty	
2	5 percent HNO_3 and 10 percent H_2O_2	100 mL
3	5 percent HNO_3 and 10 percent H_2O_2	100 mL
4	Silica gel	750g

TABLE 11. Cr(+6) TRAIN IMPINGER SYSTEM REAGENTS

Impinger Number	Reagent	Quantity
1	0.1N NaOH	100 mL
2	0.1N NaOH	100 mL
3	0.1N NaOH	100 mL
4	Silica gel	750g

One composite sample of the feed clay material was also taken and preserved for Cr(+6) analysis with 3 percent Na_2CO_3, 2 percent NaOH solution.

3.1.2 Analysis Procedures

Table 12 summarizes the number of samples collected over the three-test chromium valence state test series. Particulate load determinations from Method 5 train samples were performed at the IRF in accordance with Method 5 protocol. Chloride determinations for HCl measurement were performed on aliquots of appropriate impinger solutions via specific ion electrode analysis at the IRF.

All samples for chromium analyses, preserved as noted in Section 3.1.1, were shipped to the Acurex analytical laboratory in Mountain View, California. Total chromium analyses were performed using graphite furnace atomic absorption (AA) spectroscopy via Method 7191 for all samples except the composite feed samples (clay/organic liquid mixture). Inductively coupled argon plasma spectroscopy (ICAP) via Method 6010 was used for this sample matrix. Solid samples (probe wash, particulate, kiln ash) were digested by Method 3050 prior to analysis. Liquid samples (spike solution, blowdown) were digested by Method 3010 prior to analysis. The clay/organic liquid mixture samples were Parr bomb ashed, then digested by Method 3050 prior to analysis.

Samples for Cr(+6) analysis were digested by Method 3060. The solid samples (kiln ash and feed clay) had been preserved after collection in the method digestion solution. For these, digestion volumes were corrected and the method followed through. Digestates were analyzed by Method 7197 (chelation/extraction). Graphite furnace atomic absorption spectroscopy (in accordance with Method 7191) was used to analyze extracts from the Method 7197 procedure.

3.2 PARAMETRIC TRACE METAL TESTS

Table 13 summarizes the sampling and analysis matrix for the parametric trace metal tests.

3.2.1 Sampling Procedures

As indicated in Table 13, the incinerator flue gas was characterized at three locations: the afterburner exit, the scrubber exit, and the stack. Characterization at the afterburner and scrubber exit locations supported test objectives. Stack gas sampling was performed to ensure compliance with the IRF's operating permit.

The sampling protocols performed in the afterburner exit flue gas were designed to measure flue gas particulate load and size distribution, HCl, trace metal vapor phase and particulate emissions by particulate size range, and volatile organic hazardous constituent emissions.

Volatile organic hazardous constituent emissions were sampled using the VOST protocol. All other parameters listed above were measured in the afterburner exit flue gas using a variation of a Method 17 train. The impingers used for the afterburner exit Method 17 sampling train are noted in Table 14. This impinger train is the same as that used for total chromium in the chromium valence state tests, with the addition of a caustic impinger between the initially empty impinger and the first HNO_3/H_2O_2 impinger. After sampling, the contents of impingers 1 and 2 were combined and aliquots obtained for HCl analysis. The impinger

TABLE 12. SUMMARY OF CHROMIUM VALENCE STATE TEST SAMPLES

		Number of samples	
Sample type	**Analyte**	**Each test**	**Total**
Feed clay	Cr(+6)	—	1
Clay/organic liquid mixture	C_7H_8, C_2Cl_4, C_6H_5Cl	1	3
	Ultimate analysis	1	3
	Total Cr	1	3
Aqueous chromium spike solution	Total Cr	1	3
	Cr(+6)	1	3
Kiln ash	Total Cr	1	3
	Cr(+6)	1	3
Scrubber blowdown	Total Cr	4	12
	Cr(+6)	4	12
Afterburner exit:			
Method 5 Train A			
Probe wash	Particulate, Total Cr	1	3
Filter	Particulate, Total Cr	1	3
1st impinger	Total Cr	1	3
2nd impinger	Total Cr	1	3
3rd impinger	Total Cr	1	3
Method 5 Train B			
1st impinger	Cr(+6), Cl^-	1	3
2nd impinger	Cr(+6), Cl^-	1	3
3rd impinger	Cr(+6), Cl^-	1	3
Scrubber exit:			
Method 5 Train A			
Probe wash	Particulate, Total Cr	1	3
Filter	Particulate, Total Cr	1	3
1st impinger	Total Cr	1	3
2nd impinger	Total Cr	1	3
3rd impinger	Total Cr	1	3
Method 5 Train B			
1st impinger	Cr(+6), Cl^-	1	3
2nd impinger	Cr(+6), Cl^-	1	3
3rd impinger	Cr(+6), Cl^-	1	3
Stack gas:			
Method 5 Train			
Probe wash + filter	Particulate	1	3
1st and 2nd impinger	Cl^-	Test 3 only	1

TABLE 13. SAMPLING AND ANALYSIS MATRIX FOR THE PARAMETRIC TRACE METAL TESTS

Sample	Location	Sampling procedure	Analysis		Frequency
			Parameter	Method	
Clay/organic liquid mixture	Preparation storage drum	Trier	C_7H_8, C_2Cl_4, C_6H_5Cl	Purge and trap by Method 5030[a]; GC/FID analysis	1 composite/test
			Ultimate analysis (C, H, O, Cl)	A003[b]	1 composite/test
			Cr and Mg	Parr bomb ashing, digestion by Method 3050, ICAP analysis by Method 6010[a]	1 composite/test
			Pb	Parr bomb ashing, digestion by Method 3050, furnace AA by Method 7421[a]	1 composite/test
			Bi and Sr	Parr bomb ashing, digestion by Method 3050, AA analysis by 300 series methods[c]	1 composite/test
Aqueous metal spike solution	Kiln inlet	Composite	As and Pb	Digestion by Method 3010, furnace AA by 7000 series methods[a]	1 composite/test
			Ba, Cd, Cr, Cu, and Mg	Digestion by Method 3010, ICAP analysis by Method 6010[a]	1 composite/test
			Bi and Sr	Digestion by Method 3010[a], AA analysis by 300 series methods[c]	1 composite/test
Kiln ash	Kiln ash pit	Composite	As and Pb	Digestion by Method 3050, furnace AA by 7000 series methods[a]	1/test
			Ba, Cd, Cr, Cu, and Mg	Digestion by Method 3050, ICAP analysis by Method 6010[a]	1/test
			Bi and Sr	Digestion by Method 3050[a] AA analysis by 300 series methods[c]	1/test

(continued)

[a] (1)
[b] (3)
[c] (7)

TABLE 13. (continued)

			Analysis		
Sample	Location	Sampling procedure	Parameter	Method	Frequency
Scrubber blowdown water	Blowdown discharge	Grab/tap	As and Pb	Digestion by Method 3010, furnace AA by 7000 series methods[a]	At least 3/test
			Ba, Cd, Cr, Cu, and Mg	Digestion by Method 3010, ICAP analysis by Method 6010[a]	At least 3/test
			Bi and Sr	Digestion by Method 3010[a], AA analysis by 300 series methods[c]	At least 3/test
Flue gas	Afterburner exit	Method 17[d]	Particulate load	Method 17[d]	1/test
			Particulate size distribution	ASME PTC 28[e]	1/test
			HCl	Analysis of impinger solution for Cl by specific ion elecrode	1/test
			As and Pb	Digestion by Method 3010 or 3050, furnace AA by 7000 series methods[a] (impingers and particulate by size)	1/test
			Ba, Cd, Cr, Cu, and Mg	Digestion by Method 3010 or 3050, ICAP analysis by Method 6010[a] (impingers and particulate by size)	1/test
			Bi and Sr	Digestion by Method 3010 or 3050[a], AA analysis by 300 series methods[c] (impingers and particulate by size)	1/test
		Method 0030[a]	Volatile organic hazardous constituents	Thermal desorption, purge and trap by Method 5040[a], GC/FID analysis	3 trap pairs/ test

(continued)

---

[a] (1)

[c] (7)

[d] (5)

[e] (6)

TABLE 13. (concluded)

Sample	Location	Sampling procedure	Analysis: Parameter	Analysis: Method	Frequency
Flue gas (continued)	Scrubber exit	Method 5[d]	Particulate load	Method 5[d]	1/test
			HCl	Analysis of impinger solution for Cl by specific ion electrode	1/test
			As and Pb	Digestion by Method 3010 or 3050, furnace AA by 7000 series methods[a] (impingers and particulate)	1/test
			Ba, Cd, Cr, Cu, and Mg	Digestion by Method 3010 or 3050, ICAP analysis by Method 6010[a] (impingers and particulate)	1/test
			Bi and Sr	Digestion by Method 3010 or 3050[a], AA analysis by 300 series methods[c] (impingers and particulate)	1/test
		Method 0030[a]	Volatile organic hazardous constituents	Thermal desorption, purge and trap by Method 5040[a], GC/FID analysis	3 trap pairs/test
				Thermal desorption and trap GC/MS analysis by Method 5040[a]	3 Trap pairs, Test 4 only
Stack gas	Stack downstream of carbon bed/HEPA filter	Method 5[d]	Particulate load	Method 5[d]	1/test
			HCl	Analysis of impinger solution for Cl by specific ion electrode	
		Method 0030[a]	Volatile organic hazardous constituents	Thermal desorption, purge and trap by Method 5040[a], GC/FID analysis	3 trap pairs/test, Tests 8, 9, and 11 only

[a] (1)
[c] (7)
[d] (5)

TABLE 14. MULTIPLE METALS TRAIN IMPINGER SYSTEM REAGENTS FOR THE PARAMETRIC TRACE METAL TESTS

Impinger Number	Reagent	Quantity
1	Empty	
2	0.1 N NaOH	100 mL
3	5 percent HNO_3 and 10 percent H_2O_2	100 mL
4	5 percent HNO_3 and 10 percent H_2O_2	100 mL
5	Silica gel	750g

contents were preserved to a pH of less than 2 with HNO_3 for later metal analysis. The sampling probe was washed with acetone. The probe wash was desiccated and weighed, then resuspended in acetone for later metal analysis.

The modified Method 17 train at the afterburner exit also collected particulate in an oversized alundum thimble placed within the sampling probe. After obtaining the total particulate weight, the particulate in these samples was divided according to the terminal velocity in air using a centrifugal classifier. This device is described in the ASME Power Test Code 28 (6). Weights of the resulting eight size cuts were recorded. These samples were later recombined into four size cuts and subjected to metals analysis to provide data on metal distribution by particulate size.

The sampling protocol performed in the scrubber exit flue gas was designed to measure the same parameters measured in the afterburner exit flue gas, with the exception that particle sizing and metals analysis by particle size were not performed. Accordingly, VOST (Method 0030) sampling of the scrubber exit was performed. However, instead of a modified Method 17 train, a Method 5 train was run in the scrubber exit. The particulate levels in the scrubber exit were expected to be significantly lower than in the afterburner exit, making the collection of sufficient particulate difficult. In addition, the high water content of the scrubber exit flue gas would cause a moist and agglomerated particulate catch. Classification of this catch would likely not be representative of the actual particulate size distribution in the scrubber exit flue gas.

The impingers used in the scrubber exit Method 5 train were the same as those used in the afterburner exit Method 17 train. After sampling, the filter and probe wash (acetone) from the Method 5 train were recovered and desiccated to constant weight per the Method 5 procedure. The probe wash was then resuspended in acetone for later metal analysis. Impinger collection, aliquoting, combining, and preservation for the scrubber exit Method 5 train were exactly the same as for the afterburner exit Method 17 train discussed above.

A Method 5 train was used to sample the stack gas for each test to ensure compliance with the IRF operating permit. Particulate load and HCl levels were measured. A Method 5 train with impinger contents as noted in Table 11 was used at this location. In addition, VOST (Method 0030) sampling was performed at the stack location for Tests 8, 9, and 11 to supply continuing data on carbon bed organic component removal efficiency.

As for the chromium valence state tests, all scrubber exit Method 5 train sampling collected at least 2.8 m^3 (100 ft^3) of flue gas over a nominal 3-hour sampling period. Afterburner exit Method 17 train sampling collected at least 19.8 m^3 (700 ft^3) of flue gas over the same nominal 3-hour sampling period. This period began no less than 1/2 hour after the start of test mixture feed. Test mixture feed continued until all sampling was completed.

In addition to the flue gas sampling described above, samples of the clay/organic liquid feed mixture, the aqueous trace metal spike solution, the kiln ash, and the scrubber blowdown were collected for analysis. Composite feed samples from each drum were collected by trier sampling at three locations in the drum cross-section. Each of these composites were ultimately analyzed for toluene, tetrachloroethylene, chlorobenzene, bismuth, chromium, lead, magnesium, strontium, and ultimate composition. Samples for volatile organic analysis were collected from each drum's composite sample when the contents of that drum were first fed into the kiln. These samples were sealed in 40 mL VOA vials at that time. An aliquot of each drum's composite sample was also submitted for ultimate analysis and bismuth, chromium, lead, magnesium, and strontium analysis. Previous data suggested that clay background levels of chromium and lead might be greater than 10 percent of the final feed material (clay/organic liquid plus aqueous metal spike solution) concentration. Thus, the contribution of the clay to final concentrations of chromium and lead was measured. No data on bismuth, magnesium, and strontium concentrations in the clay existed, so analyses of the clay/organic liquid for these were also included in the analysis protocol.

A composite sample of each test's aqueous metal spike solution was also collected for trace metal analysis. These samples were preserved with HNO_3 to $pH<2$.

Blowdown sampling was performed as discussed in Section 3.1.1 for the chromium valence state tests. To reiterate, a grab (100 mL) sample was taken at the start of Method 17/5 sampling and hourly thereafter until sampling was completed. A final blowdown sample was taken at the end of the Method 17/5 sampling period. Each individual blowdown sample was preserved with HNO_3 to $pH<2$.

A composite kiln ash sample was collected from the ash pit after the completion of each test. The ash pit contents were weighed and a clean, empty ash collection drum was placed in the collection pit for the subsequent test.

3.2.2 Analysis Procedures

Table 15 summarizes the number of samples collected over the eight-test parametric trace metal program. Particulate load determinations from Methods 17 and 5 samples were performed at the IRF, in accordance with respective method procedures, prior to size classification and combination for trace metal analysis as discussed in Section 3.2.1. Chloride analyses for determining HCl emissions were performed on aliquots of appropriate impinger solution combinations via specific ion electrode analysis at the IRF.

Analysis of VOST (Method 0030) traps was performed at the IRF by thermal desorption/purge and trap GC/FID analysis. Thermal desorption/purge and trap was in

TABLE 15. SUMMARY OF PARAMETRIC TRACE METAL TEST SAMPLES

Sample type	Analyte	Number of samples	
		Each test	Total
Clay/organic liquid feed	C_7H_8, C_2Cl_4, C_6H_5Cl	1 to 3	26
	Ultimate analysis (C, H, O, Cl)	1 to 3	23
	Bi, Cr, Pb, Mg, Sr	1	8
Aqueous metal spike solution	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
Kiln ash	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
Scrubber blowdown	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	3 to 5	39
Afterburner exit flue gas:			
VOST (Method 0030)			
Sample trap pair	Volatile organic hazardous constituents	3	24
Field blank trap pair	Volatile organic hazardous constituents	1	8
Method 17 train			
<2 μm particulate	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
2 to 4 μm particulate	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
4 to 10 μm particulate	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
>10 μm particulate	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
1st and 2nd impingers	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr, Cl^-	1	8

(continued)

TABLE 15. (concluded)

		Number of samples	
Sample type	Analyte	Each test	Total
3rd impinger	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
4th impinger	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
Scrubber Exit Flue gas:			
VOST (Method 0030)			
Sample trap pair	Volatile organic hazardous constituents	3	24
Field blank trap pair	Volatile organic hazardous constituents	1	8
Method 5 train:			
Probe wash	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
Filter	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
1st and 2nd impinger	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr, Cl^-	1	8
3rd impinger	As, Ba, Cd, Cr, Pb, Bi, Cu, Mg, Sr	1	8
4th impinger	As, Ba, Cu, Cr, Pb, Bi, Cu, Mg, Sr	1	8
Stack gas:			
VOST (Method 0030)			
Sample trap pair	Volatile organic hazardous constituents	3 (Tests 5, 8, 11 only)	9
Field blank trap pairs	Volatile organic hazardous constituents	1 (Tests 5, 8, 11 only)	3
Method 5 train			
1st and 2nd impinger	Cl^-	1	8
3rd impinger	Cl^-	1	8

accordance with Method 5040. Analysis was by capillary column GC/FID. The 22 volatile organic compounds routinely determined via this method at the IRF were analyzed. These compounds are listed in Table 16.

The clay/organic liquid feed composite samples were also analyzed at the IRF for toluene, tetrachloroethylene, and chlorobenzene via purge and trap GC/FID. Purge and trap was in accordance with Method 5030. Composite clay/organic liquid feed samples were sent to Galbraith Laboratories in Knoxville, Tennessee, for ultimate analysis.

All samples for trace metal analysis were preserved as noted in Section 3.2.1, and shipped to the Acurex analytical laboratory in Mountain View, California for analysis. Arsenic and lead analyses were performed via graphite furnace atomic absorption (AA) spectroscopy: arsenic by Method 7060 and lead by Method 7421. Barium, cadmium, chromium, copper, and magnesium analyses were performed by ICAP via Method 6010. Bismuth and strontium analyses were performed by flame AA: bismuth by Method 303a and strontium by Method 326a (7). Samples were digested appropriately prior to analyses. Method 3050 was used for solid samples; Method 3010 was used for aqueous liquid samples. Clay/organic liquid samples were Parr bomb ashed (4) prior to digestion.

TABLE 16. VOLATILE ORGANIC COMPOUNDS ROUTINELY ANALYZED BY GC/FID AT THE IRF

Methylene chloride	Benzene
1,1-Dichloroethane	1,1,2-Trichloroethane
t-1,2-Dichloroethylene	Hexane
Chloroform	Bromoform
1,2-Dichloroethane	Tetrachloroethylene + Tetrachloroethane
1,1,1-Trichloroethane	Toluene
Carbon tetrachloride	Chlorobenzene
Bromodichloromethane	Ethyl benzene
1,2-Dichloropropane	1,3-Dichlorobenzene
t-1,3-Dichloropropylene	1,2-Dichlorobenzene
Trichloroethylene	1,4-Dichlorobenzene

SECTION 4

TEST RESULTS

Results from the test program are discussed in this section. The discussion is subdivided as follows: measured synthetic waste feed composition, continuous emission monitoring data, particulate and HCl emissions, chromium valence state test results, parametric trace metals test results, POHC DREs, and PIC emissions.

4.1 SYNTHETIC WASTE FEED COMPOSITION

Section 2 discussed the targeted synthetic waste feed composition planned for each test so that test program objectives could be attained. Section 3 noted that the various waste feed components were sampled during each test and analyzed to verify the actual composition of the feeds prepared. This section discusses results of the actual feed analyses.

Table 17 summarizes the clay/organic liquid mixture POHC concentrations measured for each test's composite sample and compares these to target values. The data in Table 17 confirm the close agreement between measured and target composition for all tests.

Table 18 summarizes the analyzed aqueous spike solution concentrations for Tests 1 through 3, the chromium valence state tests. Table 19 provides a similar summary for Tests 4 through 11, the parametric trace metal tests. As indicated, with one possible exception, analyzed concentrations were comparable to prepared concentrations. The exception was Test 5 in which the analyzed aqueous spike metal concentrations were lower than the prepared concentrations for all the metals. The reason for this exception is not known.

Available data on the clay matrix composition from a previous analysis indicated that the clay material contained less than 1 mg/kg of arsenic and cadmium and about 4 mg/kg copper. These values were negligible compared to the target integrated feed concentrations noted in Section 2 for these elements. These same data also indicated that the clay matrix contained about 20 mg/kg barium, 25 mg/kg lead, and 50 mg/kg chromium. These values are sufficiently high that the background clay matrix contribution to integrated feed concentrations would not be negligible. No previous data on bismuth, strontium, and magnesium concentrations were available. As a consequence, the feed clay/organic liquid mixtures for the parametric trace metals tests were analyzed for bismuth, chromium, lead, magnesium, and strontium, and a composite of the Test 1 and 2 clay/organic liquid mixture and the Test 3 mixture were analyzed for chromium to clearly determine the clay contribution to integrated feed metal concentrations. Results of these analyses are summarized in Table 20.

As indicated in Table 20, the analysis results for 10 separate feed samples were very comparable. The average values noted in the table were, thus, used to calculate the clay contribution to the integrated feed concentration for each metal. Bismuth was only analyzed for in one feed matrix sample. At the 12 ppm level noted for this sample in Table 20, the clay

TABLE 17. POHC CONCENTRATIONS IN CLAY/ORGANIC LIQUID FEED SAMPLES

Test	Test date	Weight percent in mixture			
		Toluene	Tetrachloroethylene	Chlorobenzene	Chlorine content[a]
Mixture 1 Target Composition		28.6	0	0	0
Measured composition					
1	9/29/88	27.9	0	0	0
2	9/28/88	27.9	0	0	0
4	9/14/88	23.2	0	0	0
Mixture 2 Target Composition		21.7	3.4	3.4	4
Measured composition					
5	8/25/88	16.7	3.0	3.6	3.7
6	9/16/88	20.5	3.6	3.5	4.2
7	8/30/88	19.7	3.2	3.2	3.8
8	9/07/88	17.1	3.1	3.0	3.6
9	9/09/88	16.5	2.9	2.9	3.4
10	9/20/88	22.5	3.9	3.8	4.6
Mixture 3 Target Composition		14.9	6.9	6.9	8
Measured composition					
3	9/26/88	15.9	7.5	6.7	8.5
11	9/22/88	14.6	7.1	6.9	8.3

[a] Based on measured tetrachloroethylene and chlorobenzene concentrations.

TABLE 18. AQUEOUS SPIKE SOLUTION CHROMIUM CONCENTRATIONS FOR THE CHROMIUM VALENCE STATE TESTS.

Cr valence state	Compound	Aqueous spike solution concentration (ppm Cr)					
		Test 1 (9-29-88)		Test 2 (9-28-88)		Test 3 (9-26-88)	
		Prepared	Analyzed	Prepared	Analyzed	Prepared	Analyzed
Cr(+3)	$Cr(NO_3)_3$ $9H_2O$	—	—	3400	—	3400	—
Cr(+6)	CrO_3	3400	2700	—	0.93	—	2.6
Total Cr		3400	3000	3400	3100	3400	3200

TABLE 19. AQUEOUS SPIKE SOLUTION METALS CONCENTRATIONS FOR THE PARAMETRIC TRACE METALS TEST SERIES

Metal	Compound	Prepared spike solution concentration (ppm as metal)	Analyzed spike solution concentration (ppm metal)							
			Test 4 (9/14/88)	Test 5 (8/25/88)	Test 6 (9/16/88)	Test 7 (8/30/88)	Test 8 (9/07/88)	Test 9 (9/09/88)	Test 10 (9/20/88)	Test 11 (9/22/88)
Arsenic	As_2O_3	3,230	2,800	2,300	2,900	2,400	2,400	2,300	3,600	3,300
Barium	$Ba(NO_3)_2$	2,580	2,300	1,800	2,400	2,400	2,500	2,500	2,500	2,300
Bismuth	$Bi(NO_3)_3 . 5H_2O$	9,040	10,000	6,600	11,000	7,400	9,400	11,000	11,000	11,000
Cadmium	$Cd(NO_3)_2$	650	605	410	540	530	520	540	640	540
Chromium	$Cr(NO_3)_3 . 9H_2O$	3,230	3,100	2,300	2,900	2,900	3,100	3,000	3,300	2,900
Copper	$Cu(NO_3)_2 . 3H_2O$	32,300	29,000	23,000	30,000	30,000	32,000	31,000	30,000	30,000
Lead	$Pb(NO_3)_2$	3,230	3,400	2,400	3,000	3,100	3,200	3,100	3,500	3,000
Magnesium	$Mg(NO_3)_2 . 6H_2O$	5,040	4,600	3,400	4,500	4,300	4,400	4,600	4,900	4,300
Strontium	$Sr(NO_3)_2$	16,100	18,000	11,000	15,000	14,000	15,000	16,000	19,000	17,000

TABLE 20. CLAY MATRIX METALS CONCENTRATIONS

	Concentration (mg/kg clay)[a]										
Metal	Test 1/2 composite (9-28/29-88)	Test 3 (9-26-88)	Test 4 (9-14-88)	Test 5 (8-25-88)	Test 6 (9-16-88)	Test 7 (8-30-88)	Test 8 (9-7-88)	Test 9 (9-9-88)	Test 10 (9-20-88)	Test 11 (9-22-88)	Average
Bismuth	NA[b]	NA	NA	12	NA	NA	NA	NA	NA	NA	12
Chromium	54	52	55	55	54	53	54	55	50	50	53
Lead	NA	NA	3.2	3.7	3.5	2.9	3.1	2.3	3.0	2.5	3.0
Magnesium	NA	NA	23,000	23,000	22,000	22,000	22,000	22,000	20,000	22,000	22,000
Strontium	NA	NA	33	37	36	32	33	30	33	35	34

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[a] Clay matrix residue after Parr bomb ashing.
[b] NA: not analyzed.

matrix would have a minor effect on the integrated (clay/organic liquid plus aqueous spike solution) feed bismuth concentration. Thus, other clay/organic liquid samples were not analyzed for bismuth. Barium analyses of the feed samples were inadvertently not performed. The previous analysis data (20 ppm Ba) was used to calculate the clay contribution to the integrated feed concentration for barium. A composite clay sample was analyzed for Cr(+6). The result was a Cr(+6) concentration of 2.3 ppm.

Table 21 combines the clay matrix analysis results from Table 20 with the aqueous spike solution data from Table 18 to give the chromium concentration in the integrated feed for the chromium valence state tests (Tests 1 through 3). Table 22 provides a similar summary (from the data in Tables 19 and 20) for the metals concentrations in the integrated feeds for the parametric trace metal tests (Tests 4 through 11). The clay/organic liquid feedrate data in Table 4 combined with the aqueous spike solution feedrate for each test were used to ratio individual component concentrations to give the integrated feed concentrations in Tables 21 and 22.

4.2 CONTINUOUS EMISSION MONITORING DATA

Table 23 summarizes the continuous emission monitor (CEM) data obtained for each of the tests performed. The values in the table were developed from the data recorded by the PC data acquisition system. The data in the table show that throughout the test program CO levels at the afterburner and scrubber exits remained below the detection limit of the monitors (5 ppm). CO spikes of up to 200 ppm accompanied a few of the flameouts experienced. However, as noted above, sampling was suspended during these periods. The CO/CO_2 monitor at the stack operated continuously; however, it performed erratically and had excessive drift. Reliable stack CO and CO_2 data were, therefore, not obtained. The CO/CO_2 monitor at the scrubber exit functioned well, providing reliable data for both CO and CO_2 emissions. Flue gas dilution by inleaking air between the afterburner exit and scrubber exit is evidenced by the reduction in CO_2 concentration. Finally, average NO_x concentrations at the scrubber exit ranged from 35 to 76 ppm.

TABLE 21. INTEGRATED FEED CHROMIUM CONCENTRATIONS FOR THE CHROMIUM VALENCE STATE TESTS

	Test 1 (9-29-88)	Test 2 (9-28-88)	Test 3 (9-26-88)
Total Cr Concentration (ppm)	79	84	85
Cr(+6)			
Concentration (ppm)	41	1.6	1.6
Percent of total Cr	52	2	2

TABLE 22. INTEGRATED FEED METAL CONCENTRATIONS FOR THE PARAMETRIC TRACE METALS TEST SERIES

Metal	Metal concentration (ppm)							
	Test 4 (9/14/88)	Test 5 (8/25/88)	Test 6 (9/16/88)	Test 7 (8/30/88)	Test 8 (9/7/88)	Test 9 (9/9/88)	Test 10 (9/20/88)	Test 11 (9/22/88)
Arsenic	25	23	23	24	19	19	32	33
Barium	36	33	33	39	36	36	36	37
Bismuth	98	75	95	84	85	99	110	120
Cadmium	5	4	4	5	4	4	6	5
Chromium	68	64	61	68	66	66	66	67
Copper	260	230	240	310	260	260	270	310
Lead	33	26	26	34	28	28	33	32
Magnesium	17,700	17,700	16,700	17,000	17,700	17,900	16,100	16,500
Strontium	190	140	140	170	150	160	200	200

TABLE 23. CEM DATA

Parameter	Test 1 (9/29/88)	Test 2 (9/28/88)	Test 3 (9/26/88)	Test 4 (9/14/88)	Test 5 (8/25/88)
Kiln exit					
O_2, range, percent	10.7-11.8	10.9-11.5	11.5-13.2	11.8-12.8	11.0-13.6
average, percent	11.2	11.2	11.5	12.2	11.9
Afterburner exit					
O_2, range, percent	6.6-7.9	6.4-7.7	6.3-7.7	6.1-8.0	6.0-10.2
average, percent	7.3	7.3	7.2	7.1	8.4
CO_2, range, percent	8.9-10.1	9.1-10.2	9.0-10.2	8.6-10.2	6.7-10.0
average, percent	9.5	9.5	9.5	9.5	8.2
CO, ppm	<5	<5	<5	<5	<5
Scrubber exit					
CO_2, range, percent	5.8-6.7	5.8-6.7	4.8-6.7	4.5-6.9	4.5-6.6
average, percent	6.3	6.2	6.3	6.3	5.7
CO, ppm	<5	<5	<5	<5	<5
NO_x, range, ppm	33-41	32-37	33-45	33-56	40-57
average, ppm	37	35	41	44	49
Stack					
O_2, range, percent	11.5-12.3	11.5-12.1	11.4-12.6	11.4-13.1	11.6-13.5
average, percent	11.9	11.8	11.9	11.7	12.1

(continued)

TABLE 23. (concluded)

Parameter	Test 6 (9/16/88)	Test 7 (8/30/88)	Test 8 (9/7/88)	Test 9 (9/9/88)	Test 10 (9/20/88)	Test 11 (9/22/88)
Kiln exit						
O_2, range, percent	9.0-11.0[a]	11.7-13.7	12.8-14.5	12.1-13.0	10.6-12.7	10.8-11.7
average, percent	10.6	12.4	13.6	12.6	11.4	11.4
Afterburner exit						
O_2, range, percent	5.7-7.8	7.0-8.7	3.7-7.5	6.3-7.7	6.5-8.5	6.3-8.1
average, percent	7.1	7.9	5.9[b]	7.6	7.6	7.5
CO_2, range, percent	8.7-10.4	7.6-9.4	8.9-11.8	7.7-9.0	8.5-10.0	8.5-10.0
average, percent	9.4	8.8	10.2	8.3	9.2	9.1
CO, ppm	<5	<5	<5	<5	<5	<5
Scrubber exit						
CO_2, range, percent	4.6-5.9	4.1-6.4	5.1-7.2	4.5-6.1	5.3-7.1	4.8-6.8
average, percent	5.5	5.7	6.7	5.8	6.5	6.1
CO, ppm	<5	<5	<5	<5	<5	<5
NO_x, range, ppm	45-63	35-58	59-89	32-48	43-64	39-56
average, ppm	56	50	76	42	55	48
Stack						
O_2, range, percent	11.8-13.0	11.5-14.0	10.6-11.9	12.3-13.4	13.1-14.1	11.1-12.8
average, percent	12.2	12.6	11.1	12.6	13.7	11.9

[a] CEM not in service. Data obtained using a Bacharach Fryrite O_2 analyzer.
[b] Afterburner air feedrate at fan capacity.

As mentioned in Section 1, a secondary test objective was to compare heated and unheated hydrocarbon analyzers. However, hydrocarbon emissions throughout the test program remained below the instrument detection limit of 1 ppm (except for brief periods just after a flameout). Thus, useful information for comparing analyzer performance was not obtained.

4.3 FLUE GAS PARTICULATE AND HCl

Flue gas particulate emissions were measured at the afterburner exit, scrubber exit, and stack. Particulate data are given in Table 24. HCl data are given in Table 25.

As shown in Table 24, stack particulate concentrations ranged from 29 to 58 mg/dscm at 7 percent O_2. All levels were below the hazardous waste incinerator performance standard of 180 mg/dscm at 7 percent O_2. Tests 1, 2, and 3 particulate data indicate the levels were lower at the afterburner exit than at the scrubber exit. Low afterburner values are likely a sampling artifact related to the difficulty in achieving isokinetic sampling at the afterburner exit due to the limited distance between flow disruptions. Samples taken at the afterburner exit that depend on isokinetic sampling must, therefore, be interpreted cautiously. Stack particulate emissions are generally greater than scrubber exit levels, most likely due to particulate entrainment as the gas passes through the carbon bed absorber. The greatest confidence is placed in the scrubber exit particulate concentrations where the requirements for isokinetic sampling are met.

As shown in Table 25, stack HCl levels were nondetectable at detection limits up to 6.6 mg/dscm for Tests 1 to 10. Corresponding HCl emission rates were less than 12.4 g/hr for these tests. Stack HCl levels were measurable at 12.0 mg/dscm (24.1 g/hr) for Test 11, one of the two tests at the highest feed chlorine content. All HCl stack emissions were substantially below the hazardous waste incinerator waste incinerator performance limit of 1.8 kg/hr.

Table 25 also includes information on chlorine feedrates and afterburner and scrubber exit HCl emission rates. For four of the nine tests in which flue gas HCl levels were measured, afterburner exit discharge rates agreed well with chlorine feedrates, as expected. However, for five tests, agreement was poor. The lack of correlation between chlorine feedrate and afterburner exit flue gas HCl emission rate for these five tests may be due to sampling problems associated with the flow distribution at the afterburner exit. The nonzero value measured in the afterburner exit for Test 4 (0 percent chlorine feed) may be due to hysteresis emissions of chlorides deposited during earlier tests. In any event, the afterburner exit HCl level for Test 4 is significantly lower than the values measured for the other tests.

It is interesting to note that the scrubber exit HCl levels for the two tests at the highest feed chlorine concentration (Tests 3 and 11) were above detection limits and comparable. Scrubber exit HCl levels for all but one of the other tests at lower feel chlorine concentrations were nondetectable.

4.4 TRACE METAL DISCHARGE DATA

This section discusses the distributions of the trace metals fed to the RKS in each test among the discharge streams sampled and analyzed. Distributions discussed are based on the mass flowrate of a given element in a specified discharge. This mass flowrate is calculated from the analyzed concentration of each metal in a given stream (e.g., kiln ash, flue gas particulate) and the total mass flowrate for the given stream. Appendix B includes all the laboratory analysis reports which serve as the basis for the given stream concentrations. Appendix C contains the flue gas sampling train data which includes flue gas stream flowrate information.

TABLE 24. FLUE GAS PARTICULATE DATA

		Particulate concentration (mg/dscm at 7 percent O_2)		
Test	Test date	Afterburner exit	Scrubber exit	Stack
1	9/29/88	21	31	58
2	9/28/88	23	32	42
3	9/26/88	36	37	39
4	9/14/88	273	7	44
5	8/25/88	360	7	31
6	9/16/88	162	31	27
7	8/30/88	146	23	29
8	9/07/88	50	40	40
9	9/09/88	152	12	33
10	9/20/88	414	21	47
11	9/22/88	240	51	38

TABLE 25. HCl EMISSIONS DATA

Test	Test date	Cl^- feedrate (kg/hr)	HCl concentration and emission rate					
			Afterburner exit		Scrubber exit		Stack	
			(g/dscm)	(kg/hr)	(mg/dscm)	(g/hr)	(mg/dscm)	(g/hr)
1	9/29/88	0					<5.8	<11.9
2	9/28/88	0					<4.7	<10.5
3	9/26/88	5.23	10.07	11.42	7.6	17.8	<5.6	<12.4
4	9/14/88	0	0.13	0.12	<3.9	<7.1	<6.6	<8.9
5	8/25/88	2.35	8.93	8.30	<3.4	<6.4	<5.7	<8.0
6	9/16/88	2.64	2.37	2.18	<3.9	<7.9	<5.8	<12.2
7	8/30/88	2.40	2.51	2.35	<4.2	<8.4	<5.8	<8.9
8	9/07/88	2.28	0.54	0.49	7.6	17.7	<6.6	<9.8
9	9/09/88	2.12	1.16	1.11	<4.1	<7.7	<6.4	<9.1
10	9/20/88	2.86	2.72	2.52	<2.3	<5.1	<5.8	<11.8
11	9/22/88	5.24	2.91	2.64	7.5	15.5	12.0	24.1

Table 26 summarizes the synthetic waste feedrate and kiln ash discharge flowrate data. It is interesting to note in Table 26 that the fraction of the clay matrix mass fed to the kiln accounted for by the kiln ash discharge was relatively constant at about 80 percent (range of 73 to 83 percent) for all tests. The remaining 20 percent of the clay fed represents ash entrained in the kiln exit flue gas and carried out of the kiln into the afterburner in addition to the moisture content of the clay.

The mass flowrate of a given metal removed by the venturi/packed column scrubber deserves some discussion. Prior to each test, the RKS was operated for a minimum of 18 hours fired on propane alone. Following this period, synthetic waste feed was started. Flue gas sampling was begun only after at least half an hour of waste feeding had elapsed. At the conclusion of flue gas sampling, synthetic waste feeding was stopped and the RKS was returned to propane firing. Kiln rotation continued until all ash was discharged from the kiln into the ash collection pit.

During propane firing, scrubber blowdown continued with fresh water scrubber makeup introduced to keep the scrubber liquor loop "full". As a consequence, the scrubber loop was "purged" of a previous test's metal buildup during propane-only firing such that, at the beginning of a subsequent test, the scrubber liquor was clean; it contained very little to no test trace metal content. Thus, over the time span of a given test, the recirculating scrubber liquor metal concentrations increased from near zero to some nonzero value. Further, at the end of a test, a significant portion of the metal fed to the RKS during the test which was removed by the scrubber remained in the 760 L (200 gal) scrubber liquor loop.

To account for the nonsteady scrubber loop operation over a test resulting from the above mode of operation, the scrubber blowdown sampling protocol specifically called for obtaining a scrubber blowdown sample at the beginning of a test, hourly thereafter, and at the end of a test. This allowed the buildup of metal concentrations in the scrubber liquor to be determined.

The mass flowrate of metal removed by the scrubber was then calculated as the sum of two terms. The first was that removed in the scrubber blowdown. This term was calculated from the blowdown rate noted in Table 6 and the average blowdown concentration measured from the several blowdown samples analyzed. The second term was that remaining in the 760 L (200 gal) scrubber recirculation loop at the end of a test. This term was based on the metal concentration in the last blowdown sample taken.

The following subsections summarize trace metal distributions among discharge streams for the chromium valence state and parametric trace metal tests, respectively.

4.4.1 Chromium Valence State Tests

As noted in Section 1, the objectives of this particular test series were to identify the relative distributions of chromium in the RKS discharges and to determine the fraction discharged as hexavalent chromium (Cr(+6)). The parameters varied were feed chromium valence state and feed chlorine content.

Table 27 summarizes the total chromium discharge distributions expressed as a fraction of the amount of chromium present in the feed. Almost half of the feed chromium was present as background in the feed clay absorbent. Overall mass balance closure around the system for Tests 1 and 2 determined at the two locations where the flue gas was sampled was

TABLE 26. SYNTHETIC WASTE FEEDRATES AND KILN ASH DISCHARGE RATES

		Synthetic waste feedrate, kg/hr (lb/hr)		Kiln Ash	
Test	Test data	Clay/organic liquid	Clay fraction	Discharge rate, kg/hr (lb/hr)	Fraction of clay feedrate (%)
1	9-29-88	62.6 (138.0)	45.1 (99.5)	37.4 (82.6)	83
2	9-28-88	62.0 (136.7)	44.7 (98.5)	34.9 (76.8)	78
3	9-26-88	61.3 (135.1)	42.9 (94.5)	31.3 (69.0)	73
4	9-14-88	59.4 (131.0)	45.6 (100.6)	34.7 (76.5)	76
5	8-25-88	63.5 (140.0)	48.5 (107.0)	37.8 (83.5)	78
6	9-16-88	62.6 (138.0)	45.4 (100.0)	33.6 (74.0)	74
7	8-30-88	63.4 (139.8)	46.7 (103.0)	37.8 (83.4)	81
8	9-7-88	63.4 (139.8)	48.5 (106.9)	38.3 (84.5)	79
9	9-9-88	62.5 (137.8)	48.5 (106.9)	39.8 (87.7)	82
10	9-20-88	62.8 (138.4)	43.9 (96.7)	34.7 (76.4)	79
11	9-22-88	63.5 (140.0)	45.4 (100.0)	34.5 (76.0)	76

TABLE 27. TOTAL CHROMIUM DISCHARGE DISTRIBUTIONS AND MASS BALANCE FOR THE CHROMIUM VALENCE STATE TEST SERIES

	Total Cr fraction (percent of feed)		
	Test 1 Cr(+6) feed, no Cl	Test 2 Cr(+3) feed, no Cl	Test 3 Cr(+3) feed, 8.5% feed Cl
Feed: Clay mixture	48.4	45.4	43.8
Aqueous spike	51.6	54.6	56.2
Total	100.0	100.0	100.0
Kiln ash	74.8	73.6	46.7
Afterburner exit flue gas	0.4	0.5	0.8
Around kiln and afterburber	75.2	74.1	47.5
Kiln ash	74.8	73.6	46.7
Scrubber exit flue gas	1.1	1.3	2.3
Scrubber liquor	2.5	2.3	6.4
Around kiln and scrubber	78.4	77.2	55.4

approximately 75 percent. Closure for Test 3 was poorer, 47 percent around the system with an afterburner flue gas discharge and approximately 55 percent around the system with the scrubber discharges.

Most of the chromium fed to the RKS is accounted for by the kiln ash discharge. A relatively constant, and very small, amount on percentage of feed basis was measured in the afterburner exit discharge. However, as noted in several instances in the preceding discussion, the afterburner exit flue gas sampling location is very near a major flow disturbance. Thus, it is very difficult to obtain a representative flue gas sample at this location. The lower total chromium fractions measured in the tests are probably influenced by the poor sampling characteristics of the available sampling location.

The fractions of total chromium feed accounted for in the scrubber exit flue gas and in the scrubber liquor were comparable for Tests 1 and 2 in which the feed contained no chlorine. However, these fractions were increased for Test 3 with chlorine-containing feed. This increase occurs in absolute terms despite a decrease in the overall mass balance around the kiln and scrubber for this test. This is consistent with expectations if the presence of chlorine gives rise to more volatile chromium species and more water soluble chromium species.

Table 28 summarizes the normalized total chromium discharge distributions around the RKS closed around the afterburner exit and closed around the scrubber discharges. Two discharge streams are considered with closure around the afterburner exit: the kiln ash discharge and the afterburner exit flue gas. Three discharges are considered with closure around the scrubber discharges: the kiln ash discharge, the scrubber liquor, and the scrubber exit flue gas. The distributions have been normalized with the total amount of chromium measured in the sum of the discharge streams considered for each closure case. This has the effect of normalizing discharge distributions to what they would have been if mass balance closure were 100 percent for each closure case.

The data in Table 28 emphasize the point noted above that the kiln ash discharge accounts for most of the chromium fed. In addition, as also noted above, the fraction of total chromium in the scrubber exit flue gas and the scrubber liquor were comparable for Tests 1 and 2 in which the feed contained no chlorine. No change with chromium feed valence state is seen. However, these fractions increase for Test 3 with chlorine-containing feed. As noted above, this is consistent with expectation if the presence of chlorine in the incinerator gives rise to more volatile chromium species.

Interestingly, the apparent scrubber chromium collection efficiency (scrubber liquor fraction/(scrubber liquor fraction + scrubber exit flue gas fraction)) is relatively constant at 63 to 73 percent.

Table 29 shows the distribution of total chromium between the particulate phase and the vapor/dissolved phase in the flue gas at the two locations sampled. The particulate phase fraction represents that accounted for in the probe wash and filter catch of the sampling train. The vapor/dissolved phase fraction represents that accounted for in the impingers of the sampling train. Presumably, any chromium present in the vapor phase at the sampling train probe exit would be captured and accounted for in the impingers. In addition, much of the chromium present as water soluble salts would also be collected in the impinger. The flue gas at both sampling locations contains significant water vapor. Thus, water soluble salts can be "washed" through the glass fiber filter in the sampling train and be captured in the impingers.

TABLE 28. TOTAL CHROMIUM DISCHARGE DISTRIBUTIONS FOR THE CHROMIUM VALENCE VALENCE STATE TEST SERIES

	Total Cr fraction (percent of measured)		
	Test 1 Cr(+6) feed, no Cl	Test 2 Cr(+3) feed, no Cl	Test 3 Cr(+3) feed, 8.5% feed Cl
Around kiln and afterburner			
Kiln ash	99.6	99.3	98.4
Afterburner exit flue gas	0.4	0.7	1.6
Total	100.0	100.0	100.0
Around kiln and scrubber			
Kiln ash	95.5	95.2	84.4
Scrubber exit flue gas	1.3	1.8	4.2
Scrubber liquor	3.2	3.0	11.4
Total	100.0	100.0	100.0
Apparent scrubber Cr removal efficiency	71	63	73

TABLE 29. TOTAL CHROMIUM PARTICULATE/VAPOR-DISSOLVED PHASE FLUE GAS DISTRIBUTIONS FOR THE CHROMIUM VALENCE STATE TEST SERIES

	Percent of flue gas total Cr		
	Test 1 Cr(+6) feed, no Cl	Test 2 Cr(+3) feed, no Cl	Test 3 Cr(+3) feed, 8.5% feed Cl
Afterburner exit flue gas			
Particulate	33	68	56
Vapor/dissolved phase	67	32	44
Total	100	100	100
Scrubber exit flue gas			
Particulate	57	49	79
Vapor/dissolved phase	43	51	21
Total	100	100	100

The afterburner exit data in Table 29 show that the particulate phase total chromium was highest for Test 2, the test with Cr(+3) feed and no feed chlorine; intermediate for Test 3, with the Cr(+3) feed and chlorine in the feed; and lowest for Test 1, with Cr(+6) in the feed and no feed chlorine. This would be as expected if a significant fraction of the chromium in the afterburner exit flue gas was present as soluble Cr(+6) species (e.g., $CrO_4^=$, $Cr_2O_7^=$) for the Cr(+6) feed case or as soluble chlorides in the feed chlorine case, as opposed to insoluble Cr_2O_3 in the no chlorine/Cr(+3) feed case. However, given the difficulty in obtaining a representative flue gas sample at the afterburner exit, this result should be treated with caution.

If the soluble species are more effectively removed in the scrubber, one would expect the fraction accounted for in the flue gas vapor/dissolved phase to decrease in the scrubber exit flue gas for Tests 1 and 3. This is indeed the case.

Table 30 summarizes the fractions of the total chromium analyzed as Cr(+6) in feed and discharge streams. As shown, the feed was analyzed as 52 percent Cr(+6) for Test 1, and as 2 percent Cr(+6) for Tests 2 and 3. For all tests, the kiln ash chromium was comprised of negligible amounts of Cr(+6).

The scrubber exit flue gas Cr(+6) fraction was the same (12 to 16 percent) regardless of whether or not Cr(+6) was present in the feed, in the two cases with no feed chlorine. In contrast, for the case in which the feed contained chlorine, roughly half the scrubber exit flue gas chromium was Cr(+6). This would be as expected if entrained particulate chromium from the kiln vaporized in the hotter afterburner and reacted with the flue gas chlorine to form chromyl chloride (CrO_2Cl_2), a relatively stable compound with chromium as Cr(+6).

The scrubber liquor Cr(+6) fraction for Test 1 with Cr(+6) in the feed was significantly higher than for the other two tests with only Cr(+3) in the feed. This is as expected if some of the chromium in the scrubber inlet flue gas were present as soluble Cr(+6) species ($CrO_4^=$ and $Cr_2O_7^=$ as noted above).

4.4.2 Parametric Trace Metals Tests

Experimental and laboratory data were reduced and analyzed to address the experimental objectives discussed in Section 1. Data obtained during this testing program provided information on the following:

- Distribution of metals among the kiln ash, flue gas, and scrubber liquor streams
- Afterburner exit and scrubber exit flue gas metal distributions between solid and vapor/dissolved phase
- Afterburner exit flue gas metal particulate distributions by size
- Apparent scrubber collection efficiency for each metal
- Effects of the primary test variables of kiln exit temperature, afterburner exit temperature, and feed chlorine content on metal distributions

The test results listed above are summarized and reviewed in the following subsections. To facilitate the discussion, results from all eight parametric tests are first summarized and discussed independently of the RKS operating conditions, in Section 4.4.2.1. The intent is to provide a concise summary of the relative distributions of each metal during the test program.

TABLE 30. HEXAVALENT CHROMIUM FRACTIONS FOR THE CHROMIUM VALENCE STATE TEST SERIES

	Cr(+6)/total Cr (%)		
	Test 1 **Cr(+6) feed, no Cl**	**Test 2** **Cr(+3) feed, no Cl**	**Test 3** **Cr(+3) feed, 8.5% feed Cl**
Composite feed	52	2	2
Kiln ash	0.3	0.2	0.1
Afterburner exit flue gas	102	35	4
Scrubber exit flue gas	12	16	48
Scrubber liquor	57	21	28

The relationships between the discharge distributions of the metals and the RKS operating conditions are then discussed in Section 4.4.2.2. The tables discussed in Section 4.4.2.1, therefore, contain only ranges and averages of the test results. The individual values obtained for each test are contained in the tables and figures discussed in Section 4.4.2.2.

For many samples, laboratory analysis results showed that several metals were not detected at a certain detection limit. In the data evaluation, two calculations were performed: one with the analysis result assumed to be zero, and one with the analysis result assumed to be the detection limit. In many cases, distribution conclusions were not affected. In those cases where distribution conclusions were affected, however, distributions are reported as ranges.

4.4.2.1 Average Trace Metal Discharge Distributions

As previously noted, a major objective of this test series was to identify the relative distribution of each metal among the several RKS discharge streams. Table 31 summarizes the relative distributions between the kiln ash and afterburner exit flue gas. Table 32 is a similar summary among the kiln ash, scrubber exit flue gas, and scrubber liquor. In both tables the first set of columns represents the fraction of the metal feed accounted for by the noted discharge (e.g., kiln ash, afterburner exit flue gas). The range (low, high) exhibited over the 8 tests performed and the average for all 8 tests are noted. The second set of columns represents fractions normalized to the total amount measured in the discharge streams analyzed. These normalized values represent fractions which would have resulted had mass balance closure been 100 percent. Note that the sum of the normalized values for each element for the discharge streams in each table is indeed 100.

Since the first set of columns are percent of metal feed, the kiln ash fractions are the same in both tables. However, normalized kiln ash values change between the two tables because mass balance closures experienced around the kiln ash/afterburner exit flue gas discharge differed from those experienced around the kiln ash/scrubber system discharges.

The metals are ordered in the tables by increasing average normalized kiln ash fraction, an ordering equivalent to decreasing experimentally observed average volatility. Those metals with low average normalized kiln ash fractions were more volatile (found in kiln ash at lower percentages) than metals with higher average normalized kiln ash fractions.

Cadmium was not detected in any kiln ash sample. The kiln ash fractions noted correspond to the kiln ash detection limits. The normalized cadium fractions for the other discharge streams in the tables assume cadmium was present in kiln ash at the detection limit.

Chromium is not included in Table 31. The afterburner exit flue gas sampling train contained a stainless steel condenser between the alundum thimble filter, employed to collect the large quantities of particulate needed for size fractionation, and the impinger train. As a consequence, all afterburner exit sampling train impinger solutions contained significant amounts of chromium.

The data in Tables 31 and 32 show that there are two distinct groupings of metals. Cadmium, lead, and bismuth (Table 32) tend to be relatively volatile and are not found to a major degree in the kiln ash. Barium, copper, strontium, arsenic, chromium, and magnesium were found to be more refractory; kiln ash accounted for the major fraction of these metals.

The last column in Tables 31 and 32 contains a value termed volatility temperature (8). This is the temperature at which the vapor pressure of the most volatile principal species of each

TABLE 31. SUMMARY OF METAL DISCHARGE DISTRIBUTIONS IN THE KILN ASH AND AFTERBURNER EXIT FLUE GAS FOR THE TRACE METALS PARAMETRIC TEST SERIES

	Distribution (% of metal fed)			Normalized distribution (% of total measured)			Volatility temperature[a], °C (°F)
	Kiln ash						
Metal	**Low**	**High**	**Average**	**Low**	**High**	**Average**	
Cadmium	<9.7	<17.1	<12.7	<13.7	<43.6	<28.9	216(420)
Lead	4.7	12.6	8.3	9.5	61.7	35.5	627(1160)
Bismuth	8.4	24.4	16.2	53.4	89.6	70.0	621(1150)
Copper	34.8	64.2	49.0	63.6	94.1	84.8	1116(2040)
Barium	39.2	127.7	67.0	83.3	96.7	88.6	849(1560)
Arsenic	36.3	72.0	54.8	88.1	97.4	94.6	32(90)
Strontium	25.5	64.5	43.2	99.5	99.8	99.7	1454(2650)
Magnesium	69.2	133.1	91.7	99.7	99.9	99.8	1549(2820)
	Afterburner exit flue gas						
Metal	**Low**	**High**	**Average**	**Low**	**High**	**Average**	
Cadmium	14.2	70.9	29.6	56.4	86.3	71.1	
Lead	4.7	12.6	8.3	38.3	90.5	64.5	
Bismuth	2.5	19.3	6.2	10.4	46.6	30.0	
Copper	2.8	19.5	6.6	5.9	36.4	15.2	
Barium	1.4	15.4	7.3	3.3	16.7	11.4	
Arsenic	1.6	5.5	2.1	2.6	11.9	5.4	
Strontium	0.07	0.3	0.2	0.2	0.5	0.3	
Magnesium	0.1	0.3	0.2	0.1	0.3	0.2	

[a] Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

TABLE 32. SUMMARY OF METAL DISCHARGE DISTRIBUTIONS IN THE KILN ASH SCRUBBER EXIT FLUE GAS, AND SCRUBBER LIQUOR FOR THE TRACE METALS PARAMETRIC TEST SERIES

	Distribution (% of metal fed)			Normalized distribution (% of total measured)			Volility temperature[a], °C(°F)
				Kiln ash			
Metal	**Low**	**High**	**Average**	**Low**	**High**	**Average**	
Cadmium	<9.7	<17.1	<12.7	<9.3	<29.3	<14.5	216(420)
Lead	4.7	12.6	8.3	5.8	83.7	20.1	627(1160)
Bismuth	8.4	24.4	16.2	20.9	64.8	31.6	621(1150)
Barium	39.2	127.7	67.0	68.8	86.9	76.6	849(1560)
Copper	34.0	64.2	49.0	58.0	97.6	78.6	1116(2040)
Strontium	25.5	64.5	43.2	81.9	94.3	89.3	1454(2650)
Arsenic	36.3	72.0	54.8	84.0	94.4	91.0	32(90)
Chromium	54.2	88.6	68.4	85.9	95.7	92.8	1613(2935)
Magnesium	69.2	133.1	91.7	99.2	99.6	99.4	1549(2820)
				Scrubber exit flue gas			
Metal	**Low**	**High**	**Average**	**Low**	**High**	**Average**	
Cadmium	15.7	74.4	53.2	41.9	67.5	53.5	
Lead	0.9	70.6	39.7	11.6	73.6	51.0	
Bismuth	6.0	30.4	20.6	15.7	50.7	38.4	
Barium	1.3	4.3	2.2	1.6	5.5	2.7	
Copper	0.3	19.5	9.9	0.8	33.2	15.6	
Strontium	0.5	1.6	1.0	1.1	3.7	2.1	
Arsenic	1.4	4.0	2.6	2.2	8.4	4.5	
Chromium	0.7	2.7	1.6	1.1	4.2	2.3	
Magnesium	0.03	0.3	0.1	0.03	0.2	0.1	

(continued)

[a] Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

TABLE 32. (concluded)

	Distribution (% of metal fed)			Normalized distribution (% of total measured)			Volility temperature[a], °C(°F)
	Scrubber liquor						
Metal	Low	High	Average	Low	High	Average	
Cadmium	10.8	41.1	30.4	23.2	44.9	32.0	216(420)
Bismuth	7.4	27.1	16.2	19.5	36.7	30.0	621(1150)
Lead	0.4	37.2	22.3	4.7	41.1	28.9	627(1160)
Barium	11.2	31.7	17.0	11.5	28.8	20.7	849(1560)
Copper	0.7	6.4	4.1	1.6	9.1	5.8	1116(2040)
Strontium	1.5	6.3	3.9	4.4	16.0	8.6	1454(2650)
Arsenic	1.4	3.8	2.5	2.6	8.2	4.5	32(90)
Chromium	1.9	7.7	3.4	2.3	12.2	4.9	1613(2935)
Magnesium	0.3	0.6	0.5	0.3	0.7	0.5	1549(2820)

[a] Temperature at which the vapor pressure of a principal vapor species of the metal under oxidizing conditions is 10^{-6} atm (8).

metal under oxidizing conditions is 10^{-6} atm. The lower a metal's volatility temperature is, the more volatile it is expected to be. The data in Tables 31 and 32 show a remarkable correspondence between the observed volatility and volatility temperature for all the metals tested except arsenic. That is, except for arsenic, average normalized kiln ash fraction increases more or less directly with increasing volatility temperature.

Based on volatility temperature, arsenic was expected to have been the most volatile element, and the one found at lowest percentage in the kiln ash. However, the data clearly show arsenic to be apparently refractory, remaining largely with the kiln ash. The volatility temperature for arsenic is based on the vapor pressure of As_2O_3, the most volatile arsenic species under oxidizing combustion conditions. The fact that arsenic is significantly less volatile than would be expected if As_2O_3 were the predominant arsenic species suggests that either some other, more refractory arsenic compound is preferred, or that some other chemical interaction, for example strong adsorption to the clay, occurred.

Table 33 summarizes the range and average of the percentage mass balance closures achieved for all the tests performed. Again, achieved closures around the kiln ash discharge and the afterburner exit flue gas, and around the kiln ash and scrubber discharges (flue gas and scrubber liquor), are tabulated. The data in Table 33 show that average mass balance closure ranged from 25 percent for bismuth to 88 percent for magnesium around the kiln ash/afterburner exit flue gas, with an overall average of 55 percent. Individual metal closures ranged from 11 to 140 percent.

TABLE 33. SUMMARY OF ACHIEVED METAL MASS BALANCE CLOSURE FOR THE TRACE METALS PARAMETRIC TEST SERIES

	Mass balance closure[a] (% of metal fed)					
	Closed around kiln ash discharge and afterburner exit flue gas			Closed around kiln ash discharge and scrubber discharges		
Metal	Low	High	Average	Low	High	Average
Arsenic	42	74	58	39	77	60
Barium	41	140	83	57	147	86
Bismuth	11	44	25	36	74	53
Cadmium	25	84	52	37	120	96
Chromium	—	—	—	61	94	73
Copper	47	76	57	46	79	63
Lead	12	61	31	8	96	70
Magnesium	69	98	88	70	134	92
Strontium	26	65	47	28	71	48

[a](Sum of total metal discharge rate over all discharges)/(metal feedrate).

Achieved mass balance closures were better around the kiln ash/scrubber discharge, with average metal closures ranging from 48 to 96 percent. Overall average closure was 71 percent. Individual metal closures ranged from 8 to 147 percent. Previous discussion stated that the afterburner exit flue gas sampling location is a poor one and likely does not allow a representative flue gas sample to be collected. This is the most likely explanation for the lesser closures achieved around the kiln ash/afterburner exit flue gas.

Almost all achieved mass balance closures were less than 100 percent. That is, the total amount of metal which could be accounted for in the sum of discharge streams was almost always less than the amount fed. The most likely explanation for this is that some quantity of the metal fed was deposited in slag buildup in the afterburner chamber of the RKS. A gradual accumulation of slag in the afterburner was observed over the duration of the test program. This slag was no doubt a source of metal loss.

Despite the above, the mass balance closures achieved (averaging about 65 percent) are considered good. Typical trace metal mass balance closure results from past experience for combustion sources are in the 30 to 200 percent range.

Table 34 summarizes the ranges and averages of the relative distribution of each of the metals between particulate and vapor/dissolved phases in the afterburner and scrubber exit flue gas. Again, the particulate phase fraction is defined as that analyzed in the probe wash and filter catches. The vapor/dissolved phase fraction is defined as that analyzed in the sampling train impingers. The values in Table 34 assume a metal is present in a sample at the detection limit for sample concentrations reported as less than the detection limit.

The data in Table 34 show that barium was found largely in the vapor/dissolved phase in both the afterburner exit and the scrubber exit flue gas. This suggests that the specific barium species predominant in the flue gas is quite water soluble. Bismuth and strontium were found predominantly in the vapor/dissolved phase in the afterburner exit flue gas, but were found predominantly in the particulate phase in the scrubber exit flue gas. All other metals, on average, were found predominantly in the particulate phase at both locations, although individual test conditions had opposite distributions for a few of these other metals.

Table 35 summarizes the observed range in, and average of, apparent scrubber efficiencies in removing each metal from the flue gas stream. As defined in Section 4.4.1, the apparent scrubber efficiency represents the ratio of the normalized metal fraction measured in the scrubber liquor to the sum of the normalized metal fractions measured in the scrubber liquor and scrubber exit flue gas.

The data in Table 35 show that average removal efficiencies ranged from 31 to 88 percent; the overall average for all metals was 57 percent. The metals are listed in the table in order of increasing apparent scrubber efficiency. With two exceptions, the order is generally the same as the observed metal volatility order in Tables 31 and 32. The volatile metals (those found to a lesser extent in the kiln ash, such as lead, cadmium, and bismuth) have lower average apparent scrubber collection efficiencies than do the less volatile metals (those found in the kiln ash to a greater extent, such as arsenic, chromium, strontium, and magnesium).

This is as expected. The more volatile metals vaporize in the incinerator. When the flue gas is cooled or quenched, these metals subsequently condense into either condensation nuclei, resulting in very fine fume, or onto available existing particulate. The result of this volatilization/condensation occurrence is that volatile metals become enriched in fine particulate.

TABLE 34. SUMMARY OF FLUE GAS METAL PARTICULATE/VAPOR-DISSOLVED PHASE DISTRIBUTIONS FOR THE TRACE METALS PARAMETRIC TEST SERIES

	Afterburner exit flue gas						Scrubber exit flue gas					
	Vapor/dissolved phase fraction (percent)			Particulate phase fraction (percent)			Vapor/dissolved phase fraction (percent)			Particulate phase fraction (percent)		
	Low	High	Average	Low	High	Average	Low	High	Average	Low	High	Average
Arsenic	3	53	19	47	97	81	19	68	32	32	81	68
Barium	90	99	97	1	10	3	45	90	70	10	55	30
Bismuth	63	91	77	9	37	23	7	30	18	70	93	82
Cadmium	8	43	24	57	92	76	5	18	10	82	95	90
Chromium	--	--	--	--	--	--	13	33	21	67	87	79
Copper	20	71	38	29	80	62	1	55	19	45	99	81
Lead	24	84	47	26	76	53	0.1	10	4	90	99.9	96
Magnesium	9	43	25	57	91	75	7	75	32	25	93	68
Strontium	42	75	62	25	58	38	9	68	29	32	99	71

TABLE 35. SUMMARY OF APPARENT SCRUBBER EFFICIENCY RANGES AND AVERAGES FOR THE TRACE METALS PARAMETRIC TEST SERIES

Metal	Apparent scrubber removal efficiency (%)		
	Low	High	Average
Copper	19	67	31
Lead	22	48	36
Cadmium	26	52	38
Bismuth	34	55	45
Arsenic	36	64	49
Chromium	44	87	65
Strontium	65	93	78
Magnesium	63	92	84
Barium	82	95	88

That is, their concentration on a mass percent basis ($\mu g/g$ of particulate) is higher in fine particulate than in coarse particulate. This is so because:

- Metal condensing into condensation nuclei form fume particles which are very fine
- Metal condensing onto available existing particulate are mass enriched in the available fine particulate, since condensation rates per unit particulate surface area are constant, and since the available surface area to mass is higher for fine particulate

Since the more volatile metals are expected to become enriched in fine particulate, the observation that the apparent scrubber efficiencies for the more volatile metals are lower is understandable, given that a venturi scrubber is significantly more efficient at collecting coarse particulate than fine particulate.

The two exceptions to the volatility/apparent scrubber efficiency relationship noted in Table 35 were copper and barium. The average apparent collection efficiency for barium is higher than might have been expected based on its relative volatility. However, it was noted above that barium was found predominantly in the vapor/dissolved phase in both the afterburner exit and scrubber exit flue gas streams. It was suggested that this observation would be explained if the barium compound predominating in the flue gas was quite water soluble. The observation of higher-than-expected apparent scrubber collection efficiency would be similarly explained.

An explanation is being sought as to why copper has a lower apparent scrubber collection efficiency than might be expected based on its relative volatility.

4.4.2.2 Effects of Incinerator Operating Conditions on Metal Distributions

A major objective of this test series was to identify the effects of the RKS operating conditions on the metal distributions among the incinerator discharges. As discussed in Section 2, the conditions varied in the test program were kiln exit temperature, afterburner exit temperature, and waste feed chlorine content. Previous discussion in Section 4.4.2.1 considered the 8 parameter tests performed as one set of tests, without mention of variations in test conditions. In this subsection, the data from each test are presented in a format that facilitates interpretation with respect to the primary test variables.

Table 36 summarizes the metal discharge distributions and mass balances, with the discharge distributions expressed in terms of the fraction of the metal fed. Table 37 summarizes the normalized distributions in both the kiln ash and afterburner exit flue gas discharges, and in the kiln ash and scrubber discharges. As before, in cases where some samples were reported as not containing a given metal, the distributions are reported as ranges. Limits of the ranges correspond to an assumed sample concentration of zero and the detection limit, respectively. No afterburner exit data are available for tests 5 and 10, since the alundum thimble particulate sample was compromised by excessive moisture. Also, as discussed in Section 4.4.2.1, afterburner exit flue gas chromium data are not reported.

Three groupings of tests appear in Tables 36 and 37. Each grouping corresponds to a series of tests in which one test variable (kiln exit temperature, afterburner exit temperature, or feed chlorine content) varied with the other two variables held nominally constant. The center point, which was tested in duplicate (Tests 7 and 10), is included in all groupings.

The data in Table 36 represent the individual test data comprising the ranges and averages noted in Tables 31, 32, and 33. The data in Table 37 represent the individual test data comprising the ranges and averages noted in Tables 31, 32 and 35.

Figures 5 through 13 show the variation in the normalized discharge distributions among the kiln ash, scrubber exit flue gas, and scrubber liquor for each of the test metals. Three bar charts are shown in each figure corresponding to the three groupings of tests in Tables 36 and 37. Thus, each bar chart in Figures 5 through 13 corresponds to a series of tests in which one test variable was varied, with the other two variables held nominally constant. Each figure shows the normalized discharge distributions for one of the nine test trace metals. The figures are ordered by decreasing observed metal volatility; this is the ordering of the metals used in Table 32. Thus, Figure 5 shows cadium distribution (the observed most volatile metal) data; Figure 13 shows magnesium distribution (the observed most refractory metal) data.

Normalized discharge distributions have been used in Figures 5 through 13, and will be used in the following discussion, because they remove one significant source for test to test data variability, and thereby allow clearer data interpretation. Normalized distributions would be equivalent to distributions expressed as percent of metal fed had mass balance closure been 100 percent in all cases. Use of normalized distributions, therefore, "corrects" for data variability introduced by less than perfect mass balance closure. Mass balance closure varied significantly from test to test in this test program, as it has in all past experience in measuring trace metal discharges from combustion sources. Thus, variations in metal discharge distributions expressed as percent of metal fed would be affected by both actual variations due to changes in test variables and by individual test mass balance closure. Variations in normalized distributions more closely reflect those due to changes in test variables by "forcing" 100 percent mass balance closure for each test.

TABLE 36. METAL DISCHARGE DISTRIBUTIONS AND MASS BALANCE CLOSURE FOR THE TRACE METALS PARAMETRIC TEST SERIES

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average:	1071	1088	1094	1092	875	878	873	871	874	878	873	878
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
Discharge distribution (% of metal fed)												
Arsenic												
Kiln ash	71.9	39.5	36.3	40.8	72.0	39.5	36.3	66.6	63.5	39.5	36.3	47.6
Afterburner exit flue gas	—	2.0	—	5.5	1.9	2.0	—	2.2	1.6	2.0	—	3.8
Mass balance closure around kiln/afterburner	—	41.5	—	46.3	73.9	41.5	—	68.8	65.1	41.5	—	51.4
Scrubber exit flue gas	1.9-2.3	2.1-2.7	1.1-1.5	3.6-4.0	2.5-3.0	2.1-2.7	1.1-1.5	2.7-3.2	1.2-1.4	2.1-2.7	1.1-1.5	2.2-2.5
Scrubber liquor	2.1	3.8	1.4	3.7	1.9	3.8	1.4	3.3	2.7	3.8	1.4	1.4
Mass balance closure around kiln/scrubber	76.3	46.0	39.2	48.5	76.9	46.0	39.2	73.1	67.6	46.0	39.2	51.5
Barium												
Kiln ash	43.8	64.3	55.0	76.6	76.7	64.3	55.0	127.7	39.2	64.3	55.0	52.4
Afterburner exit flue gas	—	11.7	—	15.4	10.0	11.7	—	12.1	1.4	11.7	—	7.7
Mass balance closure around kiln/afterburner	—	76.0	—	92.0	86.7	76.0	—	139.8	40.6	76.0	—	60.1
Scrubber exit flue gas	2.3	1.7	4.3	1.8	2.0	1.7	4.3	2.4	1.3	1.7	4.3	1.7
Scrubber liquor	12.9	14.7	19.4	31.7	11.2	14.7	19.4	17.1	16.5	14.7	19.4	12.7
Mass balance closure around kiln/scrubber	59.0	80.7	78.7	110.1	89.9	80.7	78.7	147.2	57.0	80.7	78.7	66.8
Bismuth												
Kiln ash	16.2	16.5	10.9	9.2	8.4	16.5	10.9	20.7	24.4	16.5	10.9	23.3
Afterburner exit flue gas	—	3.5	—	8.0	2.5	3.5	—	2.5	19.3	3.5	—	14.0
Mass balance closure around kiln/afterburner	—	20.0	—	17.2	10.9	20.0	—	23.2	43.7	20.0	—	37.3
Scrubber exit flue gas	26.1	30.4	12.7	20.3	19.1	30.4	12.7	25.6	6.0	30.4	12.7	24.7
Scrubber liquor	20.4	27.1	12.7	10.5	12.7	27.1	12.7	22.5	7.4	27.1	12.7	16.3
Mass balance closure around kiln/scrubber	62.7	74.0	36.3	40.0	40.2	74.0	36.3	68.8	37.8	74.0	36.3	64.3

(continued)

TABLE 36. (continued)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.1
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average:	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
	Discharge distribution (% of metal fed)											
Cadmium												
Kiln ash	<14.9	<11.2	<9.7	<12.9	<14.5	<11.2	<9.7	<17.1	<10.9	<11.2	<9.7	<10.0
Afterburner exit flue gas	—	20.8	—	70.9	43.1	20.8	—	24.9	14.2	20.8	—	62.8
Mass balance closure around kiln/afterburner	—	21-32	—	71-83	43-57	21-32	—	25-42	14-25	21-32	—	63-73
Scrubber exit flue gas	42.1	61.6	31.1	74.4	63.4	61.6	31.1	64.7	15.7	61.6	31.1	72.8
Scrubber liquor	41.1	36.7	33.7	37.7	26.6	36.7	33.7	36.2	10.8	36.7	33.7	25.0
Mass balance closure around kiln/scrubber	83-98	98-110	65-75	107-120	90-105	98-110	65-75	101-118	27-37	98-110	65-75	98-108
Chromium												
Kiln ash	88.6	68.9	54.9	75.3	79.9	68.9	54.9	54.2	64.2	68.9	54.9	60.9
Afterburner exit flue gas	—	—	—	—	—	—	—	—	—	—	—	—
Mass balance closure around kiln/afterburner	—	—	—	—	—	—	—	—	—	—	—	—
Scrubber exit flue gas	2.7	1.5	1.1	1.7	0.9	1.5	1.1	2.6	0.7	1.5	1.1	1.8
Scrubber liquor	2.2	2.9	7.9	2.1	2.8	2.9	7.9	4.0	1.9	2.9	7.9	3.4
Mass balance closure around kiln/scrubber	93.5	73.3	63.9	79.1	83.6	73.3	63.9	60.8	66.8	73.3	63.9	66.1
Copper												
Kiln ash	63.0	43.0	41.1	64.2	52.7	43.0	41.1	49.3	44.8	43.0	41.1	34.0
Afterburner exit flue gas	—	4.0	—	11.7	8.5	4.0	—	5.9	2.8	4.0	—	19.5
Mass balance closure around kiln/afterburner	—	47.0	—	75.9	61.2	47.0	—	55.2	47.6	47.0	—	53.5
Scrubber exit flue gas	9.6	8.6	9.5	11.0	10.1	8.6	9.5	10.5	0.3	8.6	9.5	19.5
Scrubber liquor	6.4	5.1	3.3	2.7	3.6	5.1	3.3	5.9	0.7	5.1	3.3	5.2
Mass balance closure around kiln/scrubber	79.0	56.7	53.9	77.9	66.4	56.7	53.9	65.7	45.8	56.7	53.9	58.7

(continued)

TABLE 36. (concluded)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average:	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
					Discharge distribution (% of metal fed)							
Lead												
Kiln ash	11.4	12.5	5.0	8.1	4.7	12.5	5.0	12.6	6.7	12.5	5.0	5.7
Afterburner exit flue gas	—	7.7	—	31.4	26.2	7.7	—	11.2	5.7	7.7	—	55.1
Mass balance closure around kiln/afterburner	—	20.2	—	39.5	30.9	20.2	—	23.8	12.4	20.2	—	60.8
Scrubber exit flue gas	45.5	40.9	18.4	51.6	48.8	40.9	18.4	41.1	0.9	40.9	18.4	70.6
Scrubber liquor	33.3	30.3	13.2	17.2	27.0	30.3	13.2	37.2	0.4	30.3	13.2	19.6
Mass balance closure around kiln/scrubber	90.2	83.7	36.6	76.9	80.5	83.7	36.6	90.9	8.0	83.7	36.6	95.9
Magnesium												
Kiln ash	133.1	97.9	75.3	90.4	96.2	97.9	75.3	69.2	89.1	97.9	75.3	82.6
Afterburner exit flue gas	—	0.1	—	0.3	0.3	0.1	—	0.1	0.1	0.1	—	0.2
Mass balance closure around kiln/afterburner	—	98.0	—	90.7	96.5	98.0	—	69.3	89.2	98.0	—	82.8
Scrubber exit flue gas	0.3	0.05	0.1	0.05	0.06	0.05	0.1	0.07	0.03	0.05	0.1	0.07
Scrubber liquor	0.5	0.6	0.4	0.4	0.6	0.6	0.4	0.5	0.3	0.6	0.4	0.4
Mass balance closure around kiln/scrubber	133.9	98.6	75.8	90.9	96.9	98.6	75.8	69.8	89.4	98.6	75.8	83.1
Strontium												
Kiln ash	32.6	56.7	34.1	64.1	64.5	56.7	34.1	34.8	25.5	56.7	34.1	33.1
Afterburner exit flue gas	—	0.3	—	0.2	0.1	0.3	—	0.1	0.07	0.3	—	0.1
Mass balance closure around kiln/afterburner	—	57.0	—	64.3	64.6	57.0	—	34.9	25.6	57.0	—	33.2
Scrubber exit flue gas	0.5	1.0	1.3	1.2	1.0	1.0	1.3	1.6	0.6	1.0	1.3	0.7
Scrubber liquor	6.3	3.3	2.5	5.9	3.1	3.3	2.5	6.1	1.5	3.3	2.5	2.8
Mass balance closure around kiln/scrubber	39.4	61.0	37.9	71.2	68.6	61.0	37.9	42.5	27.6	61.0	37.0	36.6

TABLE 37. NORMALIZED METAL DISCHARGE DISTRIBUTIONS AND APPARENT SCRUBBER COLLECTION EFFICIENCY FOR THE TRACE METALS PARAMETRIC TEST SERIES

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
	Normalized discharge distribution (%)											
Arsenic												
Kiln ash	–	95.1	–	88.1	97.3	95.1	–	96.9	97.4	95.1	–	92.7
Afterburner exit flue gas	–	4.9	–	11.9	2.7	4.9	–	3.1	2.6	4.9	–	7.3
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	94.4	86.1	92.3	84.0	93.6	86.1	92.3	91.2	93.9	86.1	92.3	92.4
Scrubber exit flue gas	2.1-2.9	3.8-5.8	2.3-4.1	6.8-8.4	2.6-3.8	3.8-5.8	2.3-4.1	3.0-4.3	1.7-2.2	3.8-5.8	2.3-4.1	4.0-4.8
Scrubber liquor	2.7	8.2	3.6	7.6	2.6	8.2	3.6	4.6	3.9	8.2	3.6	2.7
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	48-57	59-69	47-62	47-53	40-50	59-69	47-62	52-61	64-70	59-69	47-62	36-41
Barium												
Kiln ash	–	84.6	–	83.3	88.4	84.6	–	91.6	96.7	84.6	–	87.2
Afterburner exit flue gas	–	15.4	–	16.7	11.6	15.4	–	8.4	3.3	15.4	–	12.8
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	74.3	79.6	69.9	69.6	85.2	79.6	69.9	86.9	68.8	79.6	69.9	78.6
Scrubber exit flue gas	3.8	2.2	5.5	1.6	2.2	2.2	5.5	1.6	1.6-2.4	2.2	5.5	2.4
Scrubber liquor	21.9	18.2	24.7	28.8	12.5	18.2	24.7	11.5	28.8	18.2	24.7	19.0
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	85	89	82	95	85	89	82	88	92	89	82	89
Bismuth												
Kiln ash	–	82.6	–	53.4	76.3	82.6	–	89.6	56.0	82.6	–	62.4
Afterburner exit flue gas	–	17.4	–	46.6	23.7	17.4	–	10.4	44.0	17.4	–	37.6
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	25.8	22.2	30.0	22.9	20.9	22.2	30.0	30.1	64.8	22.2	30.0	36.3
Scrubber exit flue gas	41.5	41.1	35.2	50.7	47.4	41.1	35.2	37.1	15.7	41.1	35.2	38.4
Scrubber liquor	32.6	36.7	34.7	26.3	31.6	36.7	34.7	32.8	19.5	36.7	34.7	25.4
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	44	47	50	34	40	47	50	47	55	47	50	40

(continued)

TABLE 37. (continued)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
	Normalized discharge distribution (%)											
Cadmium												
Kiln ash	–	<35.1	–	<15.3	<25.1	<35.1	–	<40.7	<43.6	<35.1	–	<13.7
Afterburner exit flue gas	–	>64.9	–	>84.7	>74.9	>64.9	–	>59.3	>56.4	>64.9	–	>86.3
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	<15.2	<10.3	<12.9	<10.7	<13.9	<10.3	<12.9	<14.5	<29.3	<10.3	<12.9	<9.3
Scrubber exit flue gas	43-49	56-61	42-45	62-69	61-69	56-61	42-45	55-62	42-54	56-61	42-45	68-74
Scrubber liquor	42-51	34-39	45-55	27-31	25-31	34-39	45-55	31-38	29-46	34-39	45-55	23-26
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	51	39	52	31	31	39	52	38	46	39	52	26
Chromium												
Kiln ash	–	–	–	–	–	–	–	–	–	–	–	–
Afterburner exit flue gas	–	–	–	–	–	–	–	–	–	–	–	–
Total	–	–	–	–	–	–	–	–	–	–	–	–
Kiln ash	94.7	94.1	85.9	95.3	95.5	94.1	85.9	89.3	95.7	94.1	85.9	92.1
Scrubber exit flue gas	3.0	2.0	1.9	2.1	1.1	2.0	1.9	4.2	1.4	2.0	1.9	2.8
Scrubber liquor	2.3	3.9	12.2	2.6	3.4	3.9	12.2	6.6	2.8	3.9	12.2	5.1
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	44	66	87	56	75	66	87	61	67	66	87	64
Copper												
Kiln ash	–	90.8	–	84.7	86.0	90.8	–	89.4	94.1	90.8	–	63.6
Afterburner exit flue gas	–	9.2	–	15.3	14.0	9.2	–	10.6	5.9	9.2	–	36.4
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	84.2	75.8	76.2	82.3	79.2	75.8	76.2	75.1	97.6	75.8	76.2	58.0
Scrubber exit flue gas	12.9	15.1	17.8	14.1	15.2	15.1	17.8	16.0	0.8	15.1	17.8	33.2
Scrubber liquor	3.0	9.1	5.9	3.6	5.6	9.1	5.9	8.9	1.6	9.1	5.9	8.8
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	19	38	25	21	27	38	25	36	67	38	25	21

(continued)

TABLE 37. (concluded)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
						Normalized discharge distribution (%)						
Lead												
Kiln ash	–	61.7	–	20.3	15.2	61.7	–	52.8	53.7	61.7	–	9.5
Afterburner exit flue gas	–	38.3	–	79.7	84.8	38.3	–	47.2	46.3	38.3	–	90.5
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	12.6	15.0	13.7	10.4	5.8	15.0	13.7	13.8	83.7	15.0	13.7	6.0
Scrubber exit flue gas	50.4	48.9	50.2	67.2	60.6	48.9	50.2	45.0	11.6	48.9	50.2	73.6
Scrubber liquor	37.0	36.1	36.0	22.4	33.6	36.1	36.0	41.1	4.7	36.1	36.0	20.3
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	42	42	42	25	36	42	42	48	29	42	42	22
Magnesium												
Kiln ash	–	99.9	–	99.7	99.7	99.9	–	99.9	99.9	99.9	–	99.8
Afterburner exit flue gas	–	0.1	–	0.3	0.3	0.1	–	0.1	0.01	0.1	–	0.2
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	99.4	99.3	99.3	99.5	99.3	99.3	99.3	99.2	99.6	99.3	99.3	99.4
Scrubber exit flue gas	0.2	0.1	0.2	0.1	0.1	0.1	0.2	0.1	0.03	0.1	0.2	0.1
Scrubber liquor	0.4	0.6	0.5	0.4	0.6	0.6	0.5	0.7	0.36	0.6	0.5	0.5
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	63	92	75	89	91	92	75	88	91	92	75	85
Strontium												
Kiln ash	–	99.5	–	99.7	99.8	99.5	–	99.6	99.7	99.5	–	99.7
Afterburner exit flue gas	–	0.5	–	0.3	0.2	0.5	–	0.4	0.3	0.5	–	0.3
Total	–	100	–	100	100	100	–	100	100	100	–	100
Kiln ash	82.9	93.0	89.8	90.1	94.3	93.0	89.8	81.9	91.8	93.0	89.8	90.7
Scrubber exit flue gas	1.1	1.7	3.5	1.6	1.3	1.7	3.5	3.7	2.5	1.7	3.5	1.6
Scrubber liquor	16.0	5.3	6.6	8.3	4.4	5.3	6.6	14.4	5.7	5.3	6.6	7.7
Total	100	100	100	100	100	100	100	100	100	100	100	100
Apparent scrubber efficiency	93	76	65	84	77	76	65	80	69	76	65	83

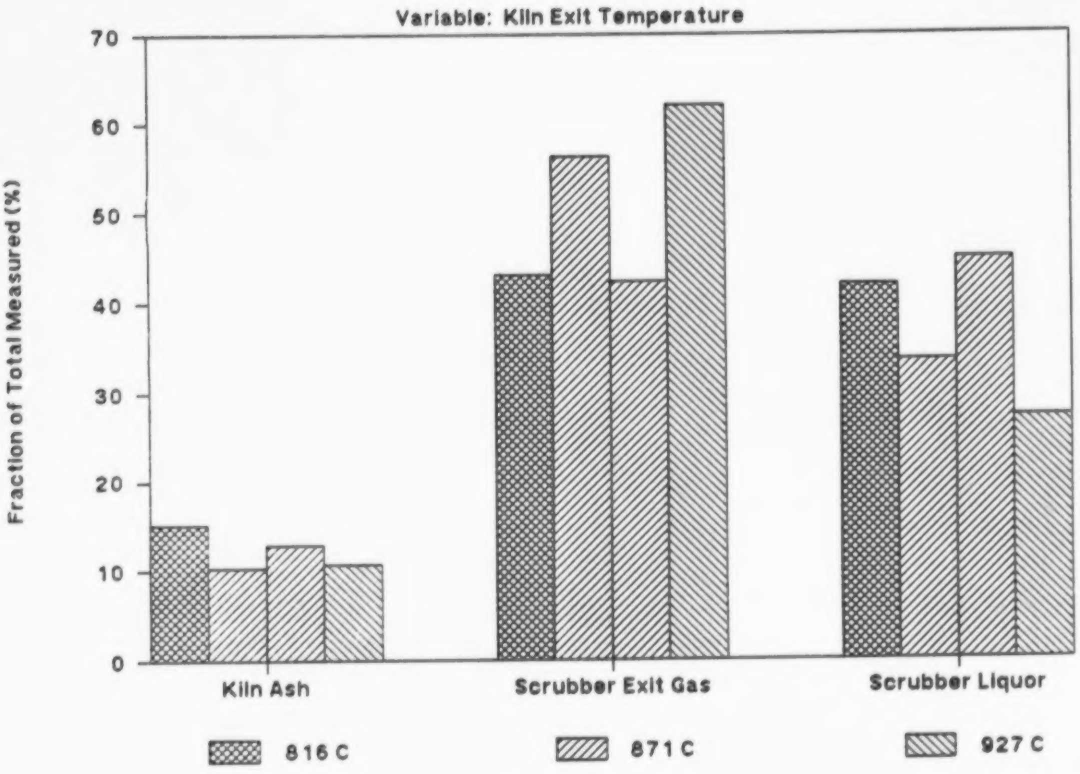


Figure 5a. **Cadmium discharge distributions for the parametric trace metal tests: effect of kiln temperature.**

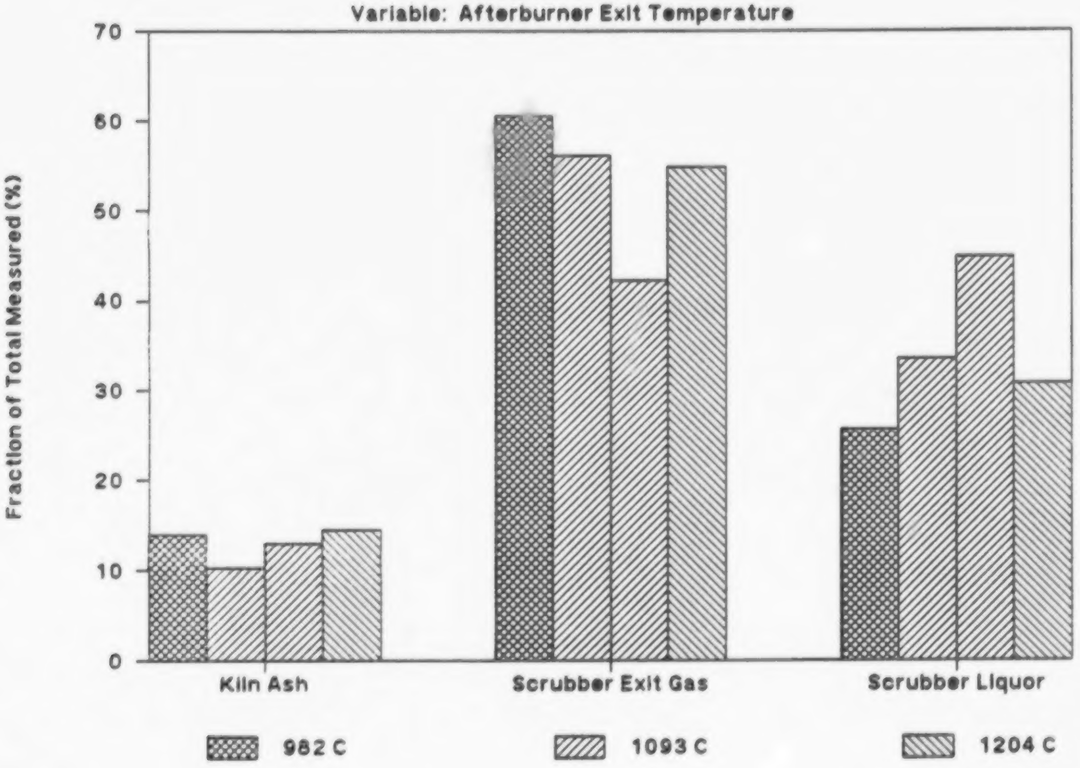


Figure 5b. Cadmium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

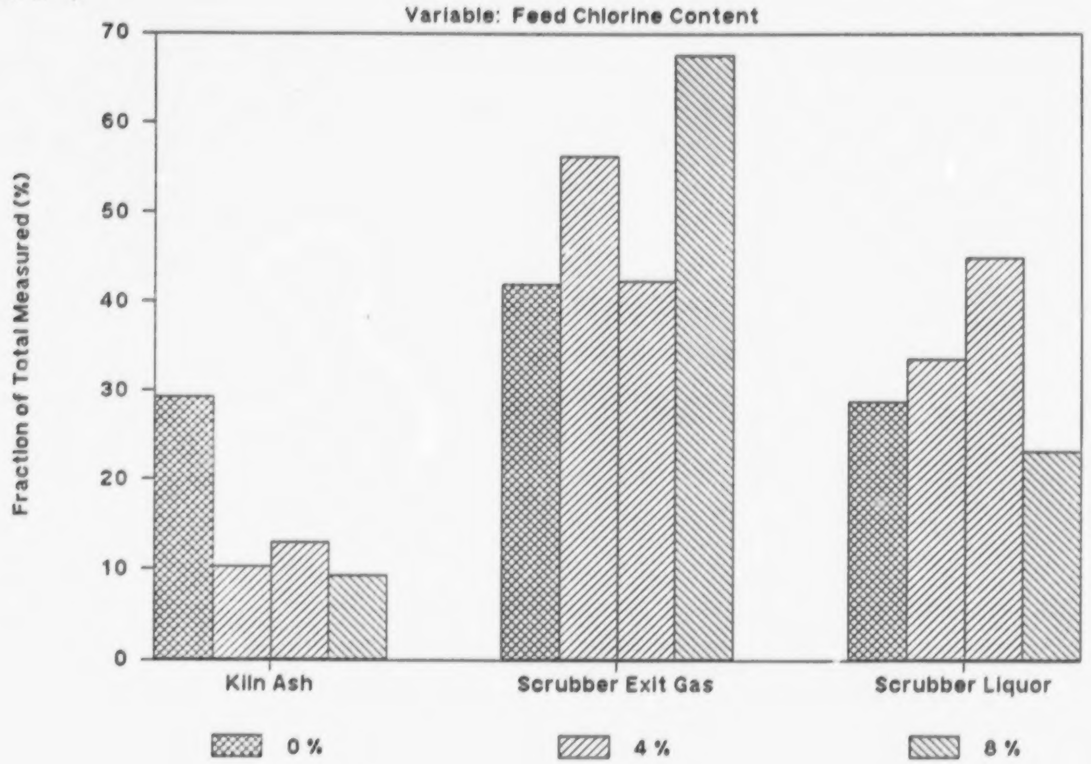


Figure 5c. **Cadmium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.**

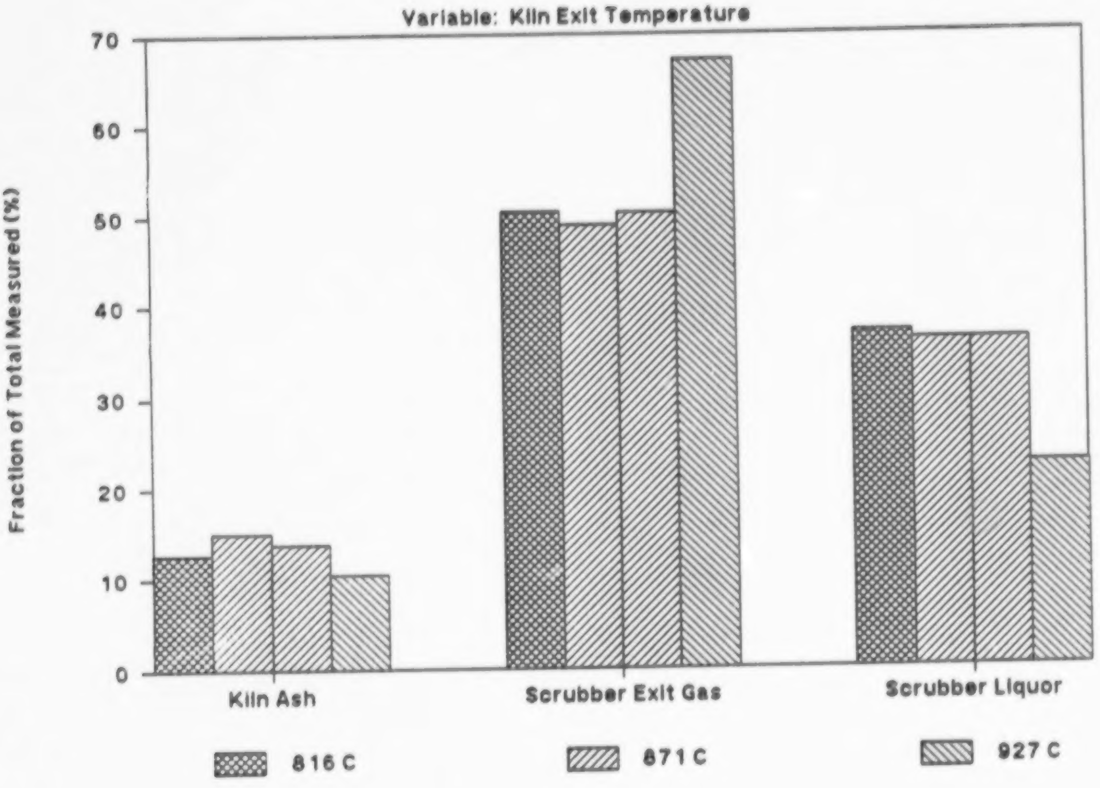


Figure 6a. Lead discharge distributions for the parametric trace metal tests: effect of kiln temperature.

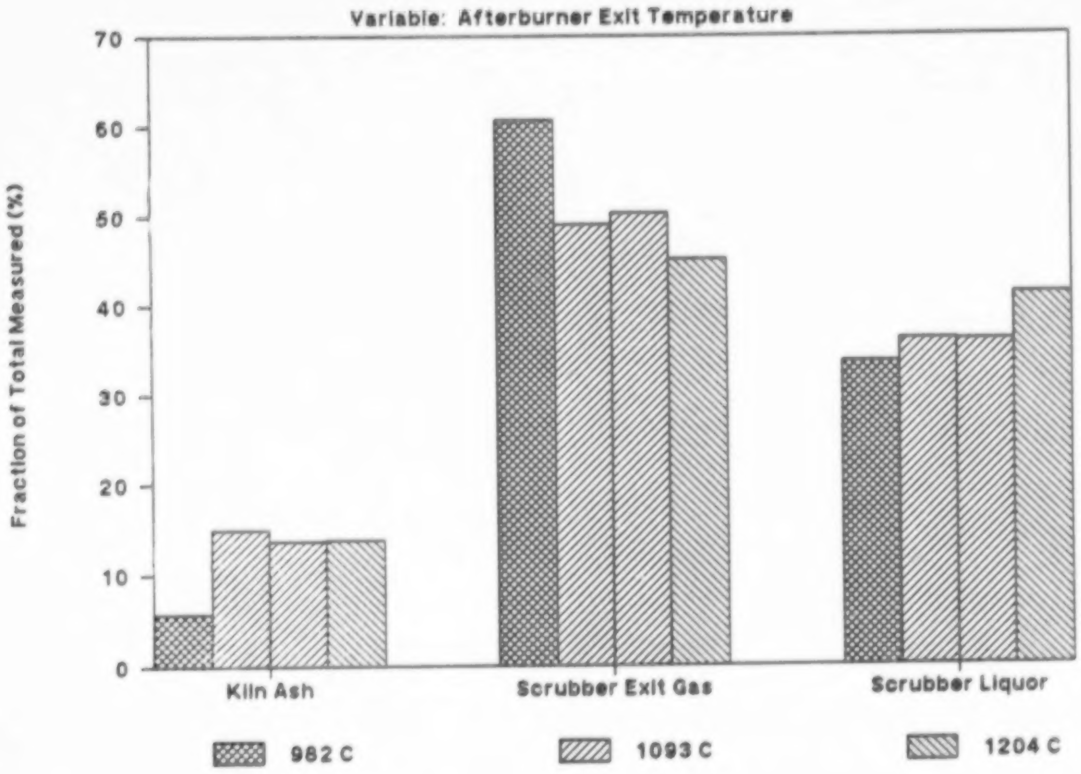


Figure 6b. **Lead discharge distributions for the parametric trace metal tests: effect of afterburner temperature.**

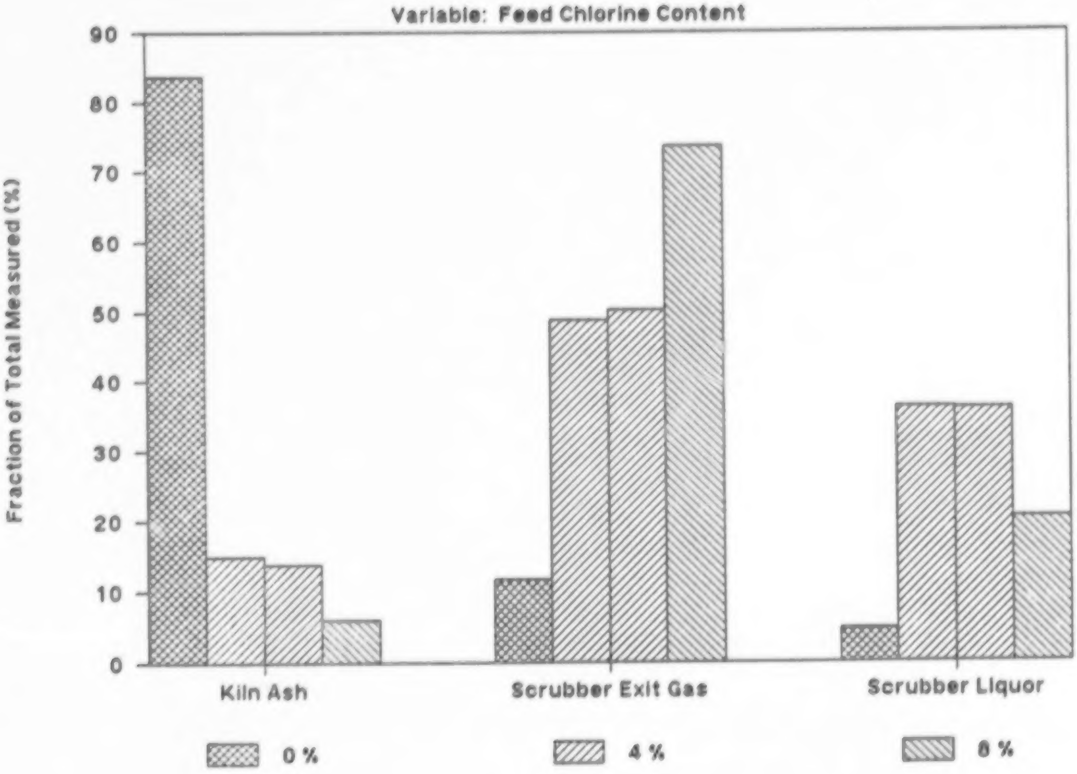


Figure 6c. Lead discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

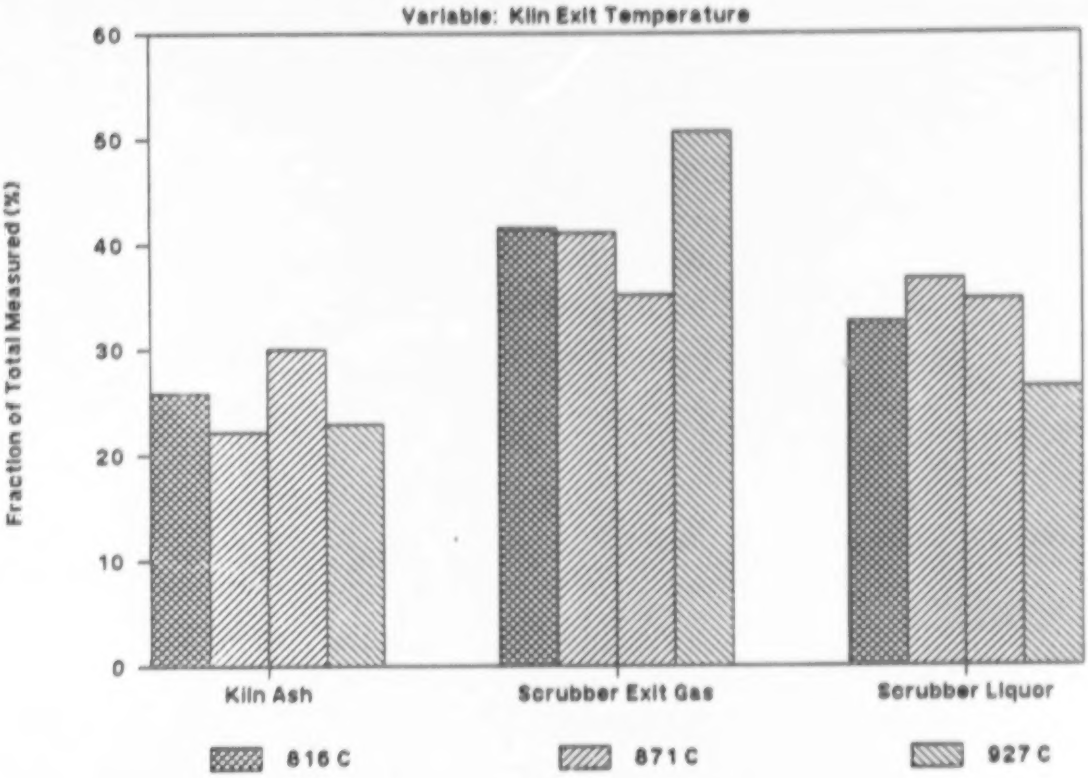


Figure 7a. Bismuth discharge distributions for the parametric trace metal tests: effect of kiln temperature.

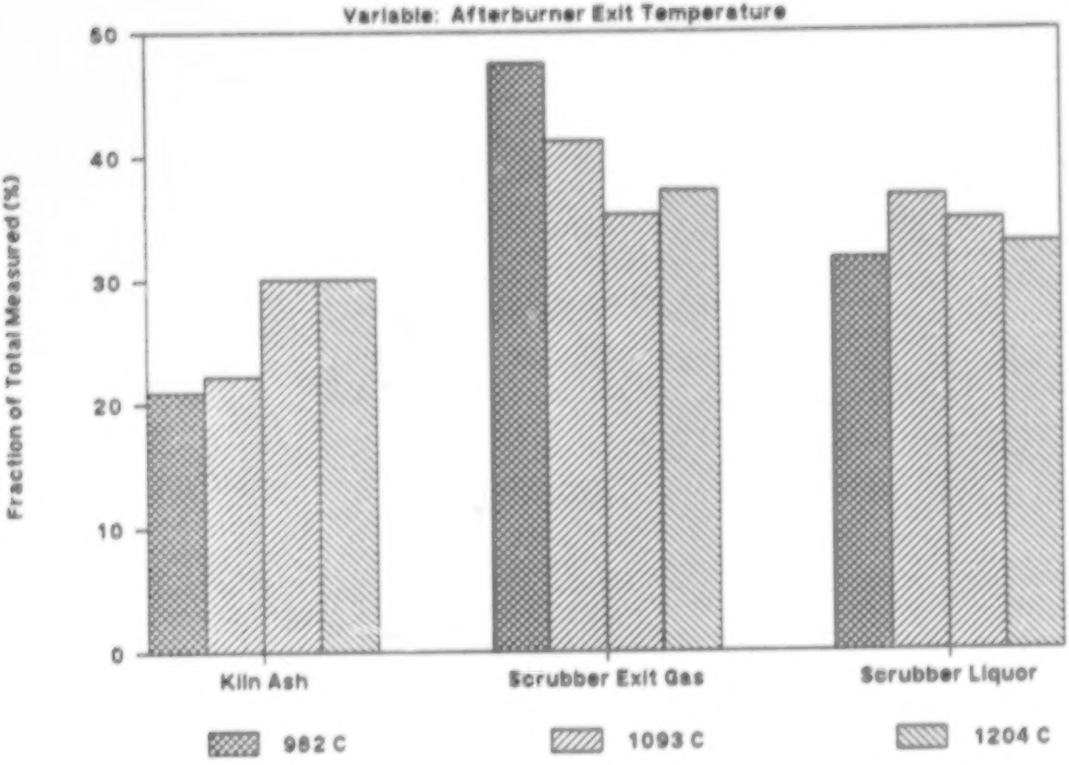


Figure 7b. **Bismuth discharge distributions for the parametric trace metal tests: effect of afterburner temperature.**

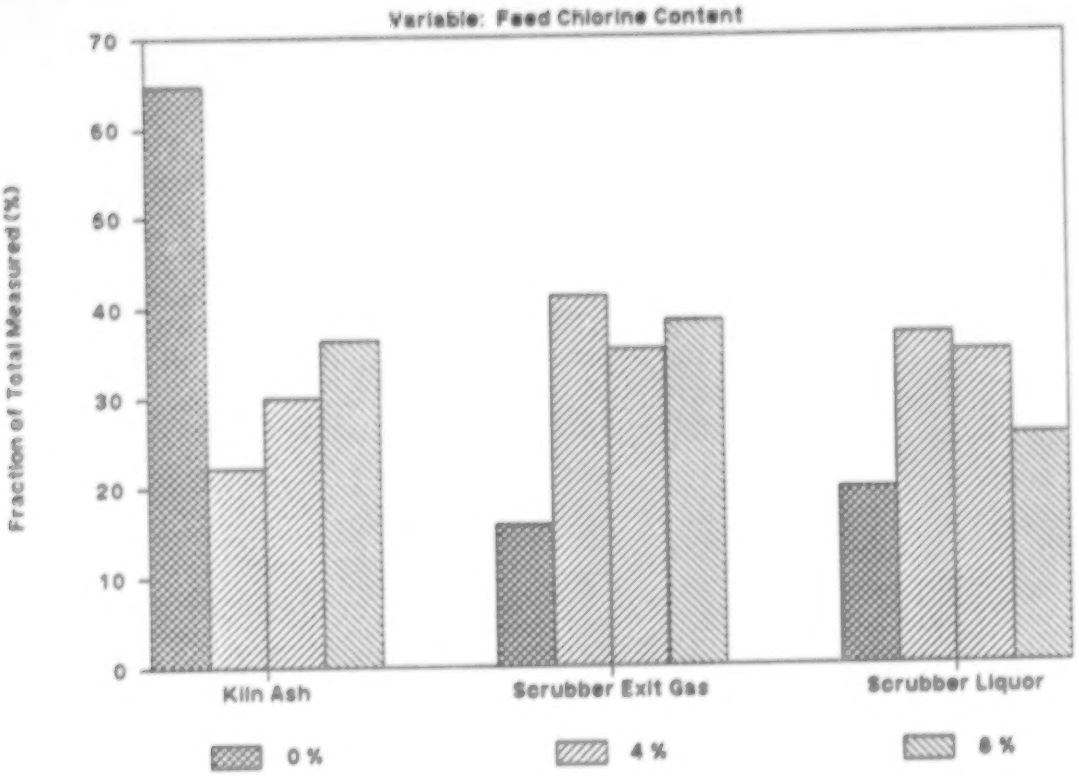


Figure 7c. Bismuth discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

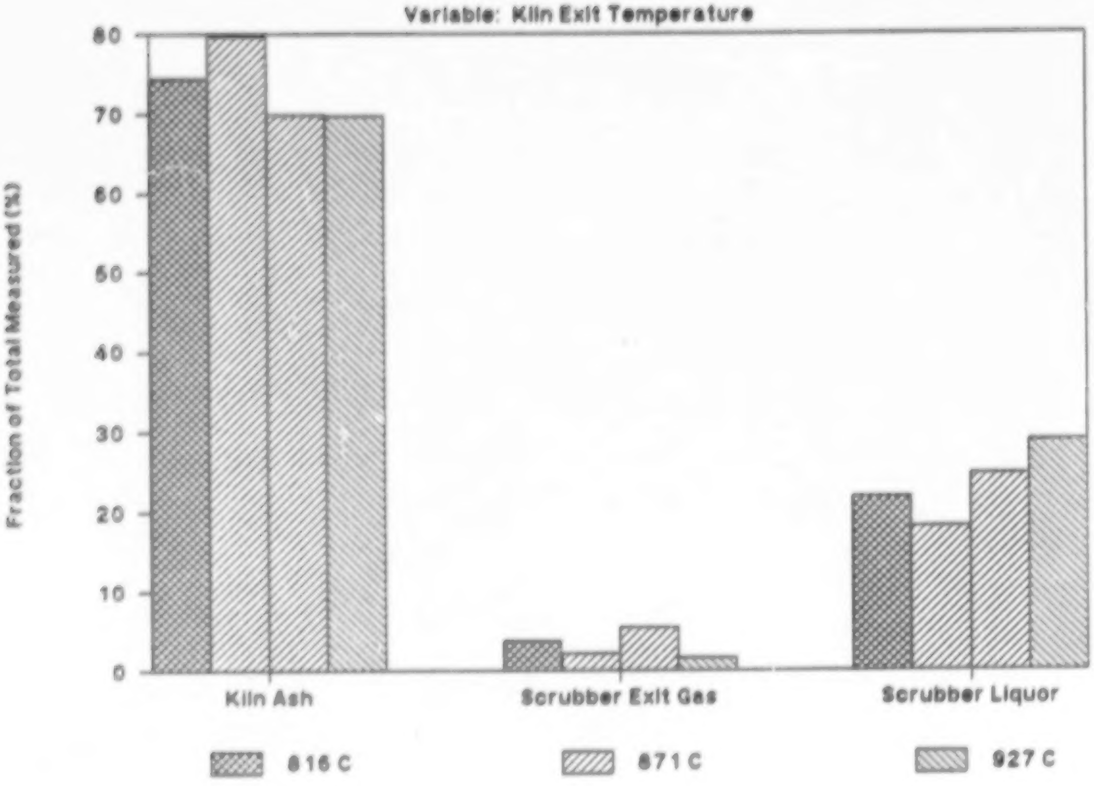


Figure 8a. Barium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

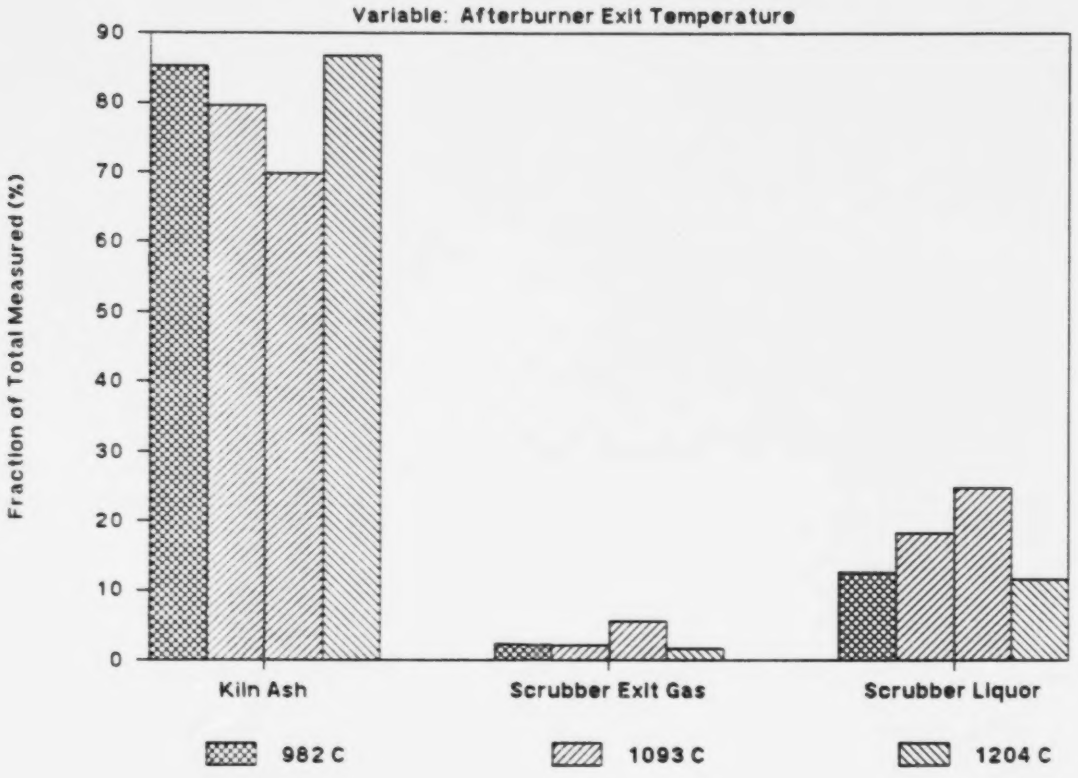


Figure 8b. Barium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

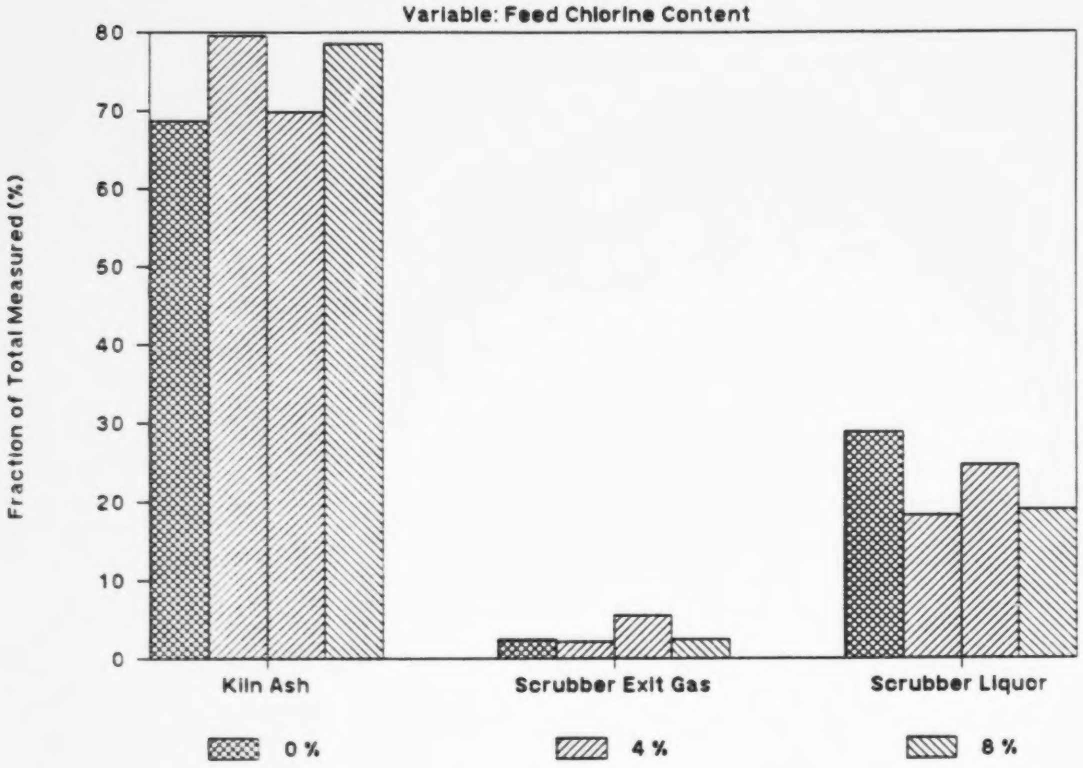


Figure 8c. Barium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

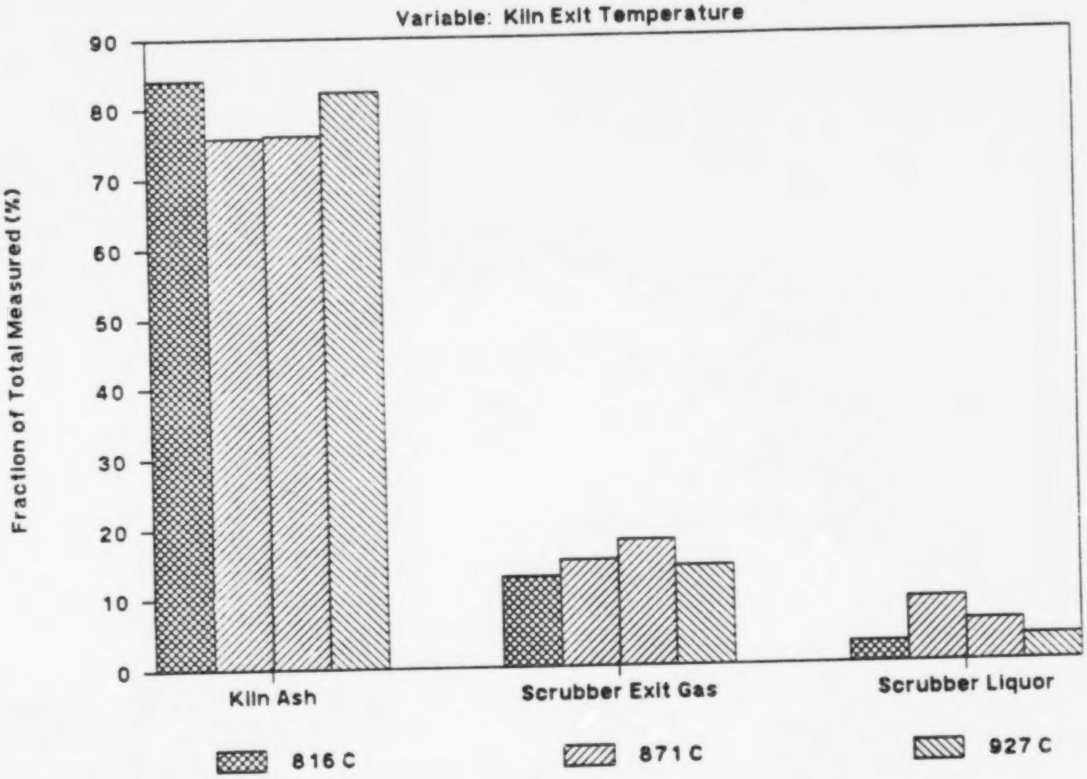


Figure 9a. Copper discharge distributions for the parametric trace metal tests: effect of kiln temperature.

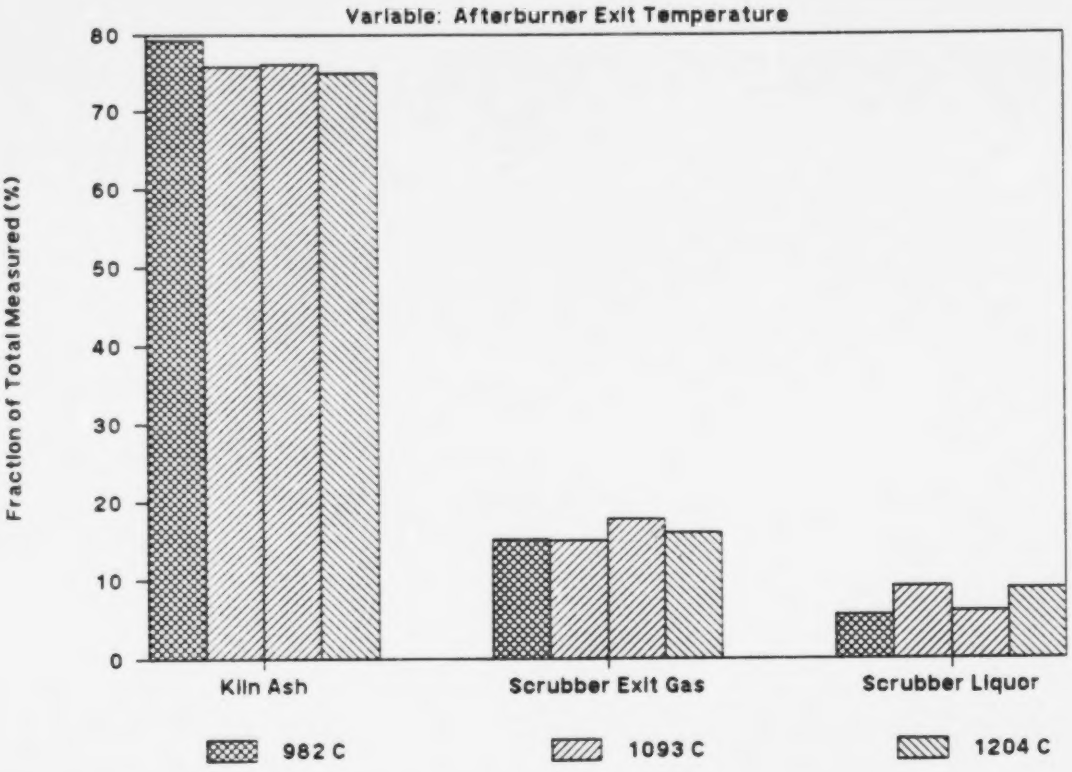


Figure 9b. **Copper discharge distributions for the parametric trace metal tests: effect of afterburner temperature.**

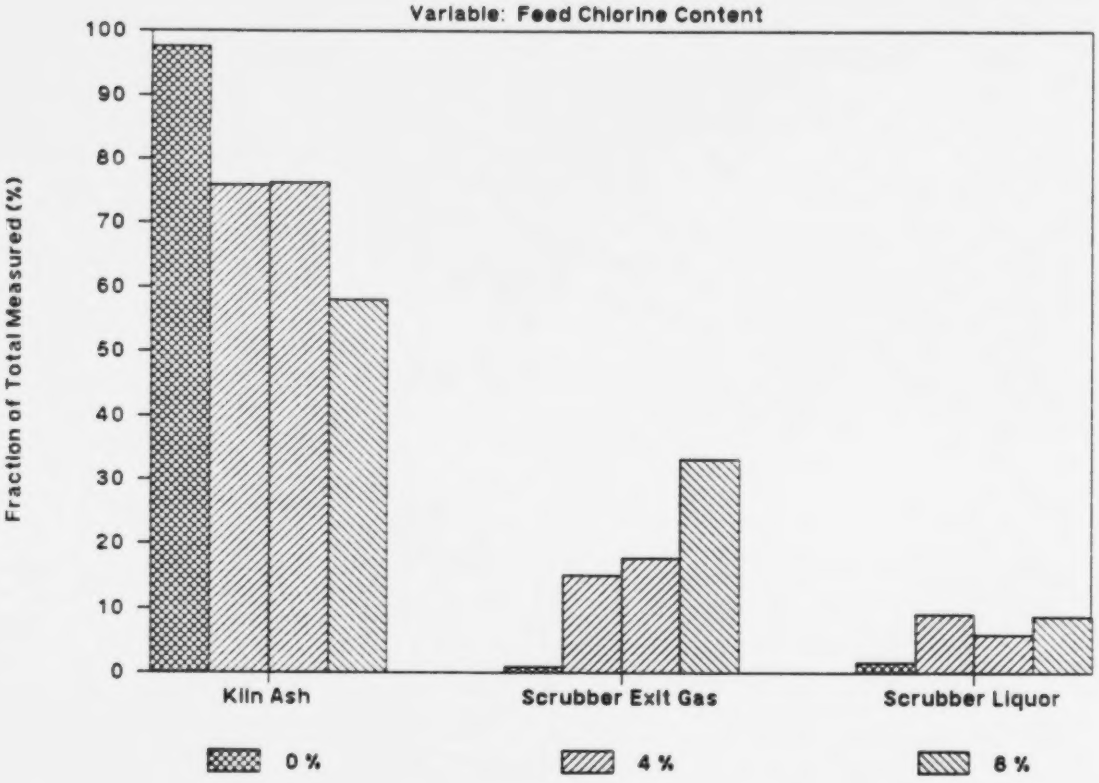


Figure 9c. **Copper discharge distributions for the parametric trace metal tests: effect of feed chlorine content.**

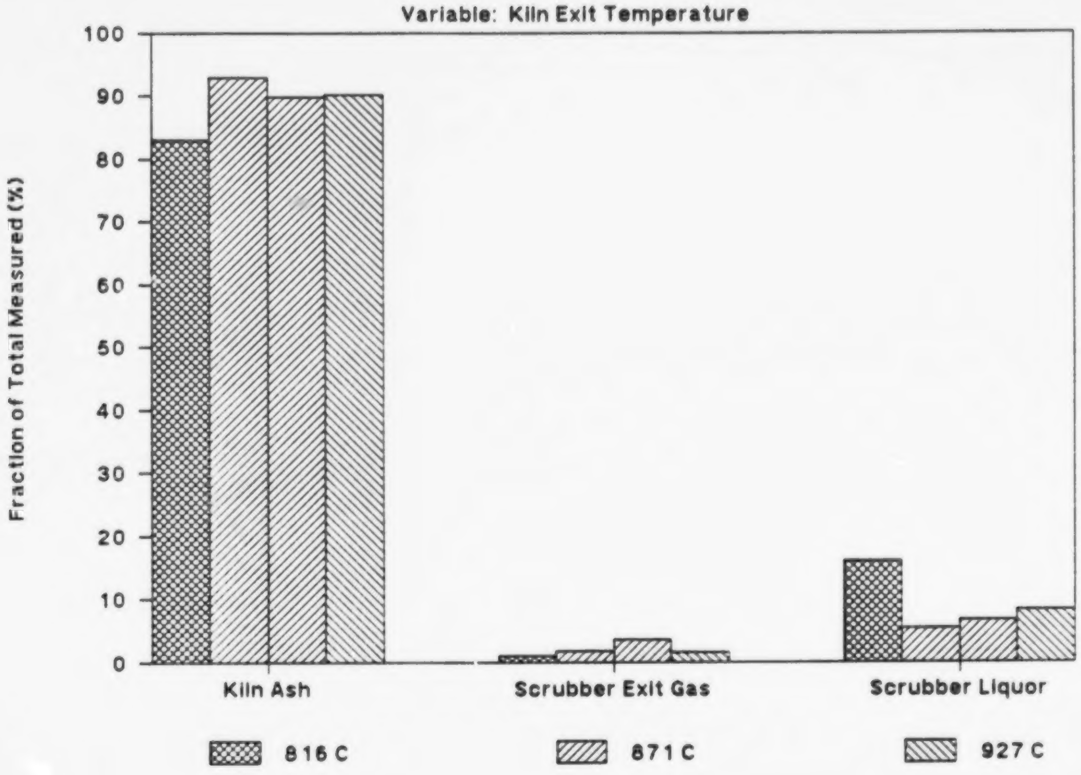


Figure 10a. **Strontium discharge distributions for the parametric trace metal tests: effect of kiln temperature.**

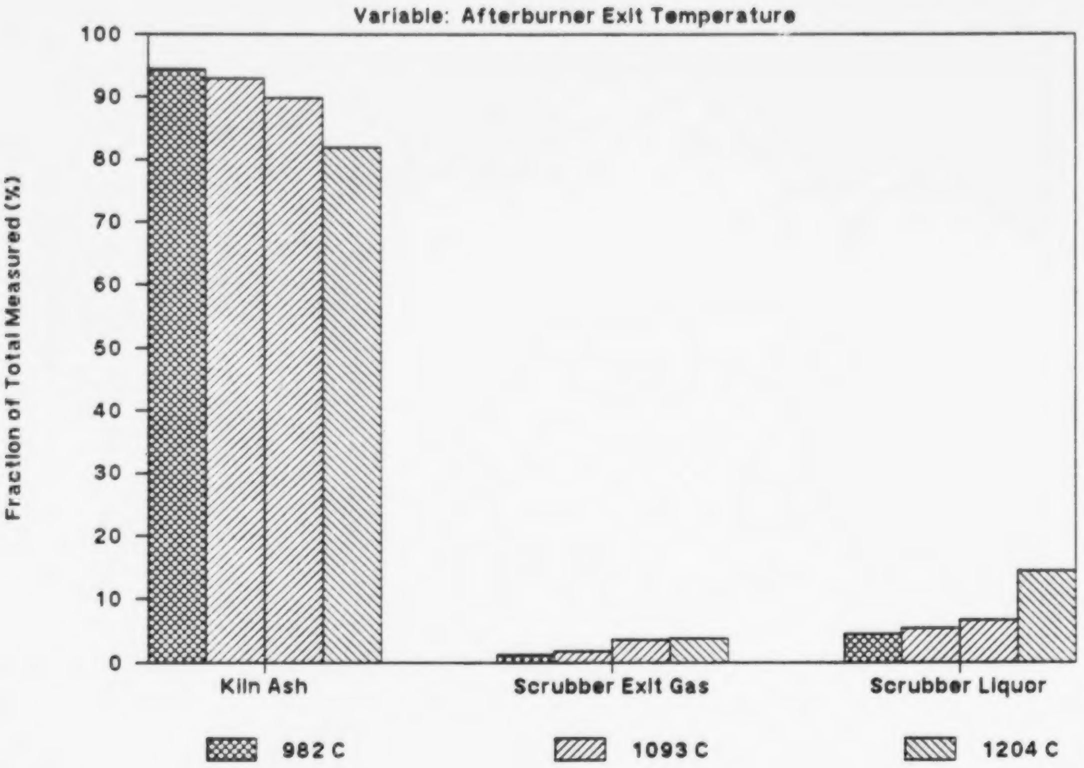


Figure 10b. **Strontium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.**

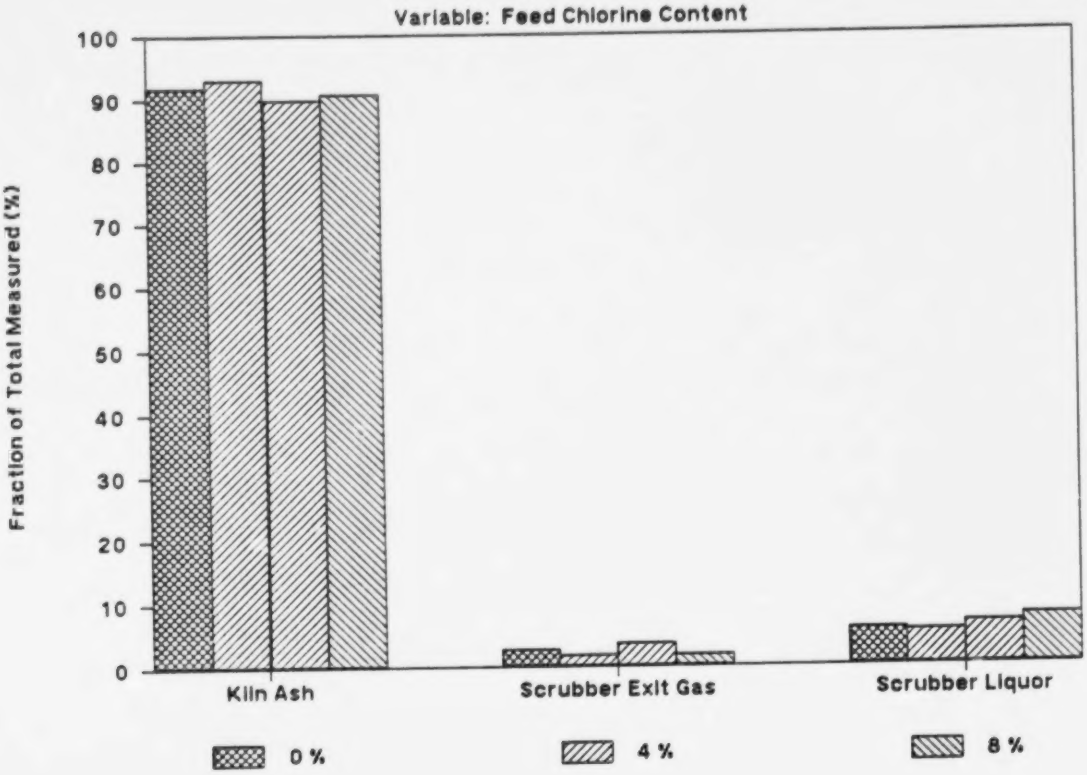


Figure 10c. **Strontium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.**

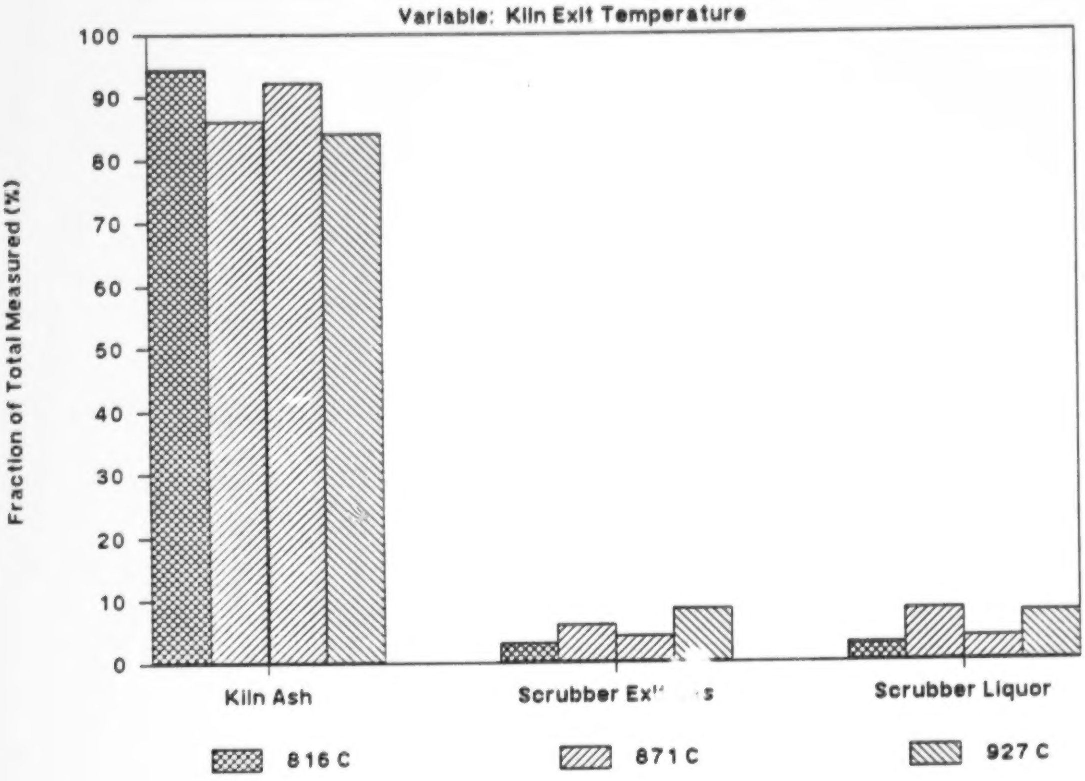


Figure 11a. Arsenic discharge distributions for the parametric trace metal tests: effect of kiln temperature.

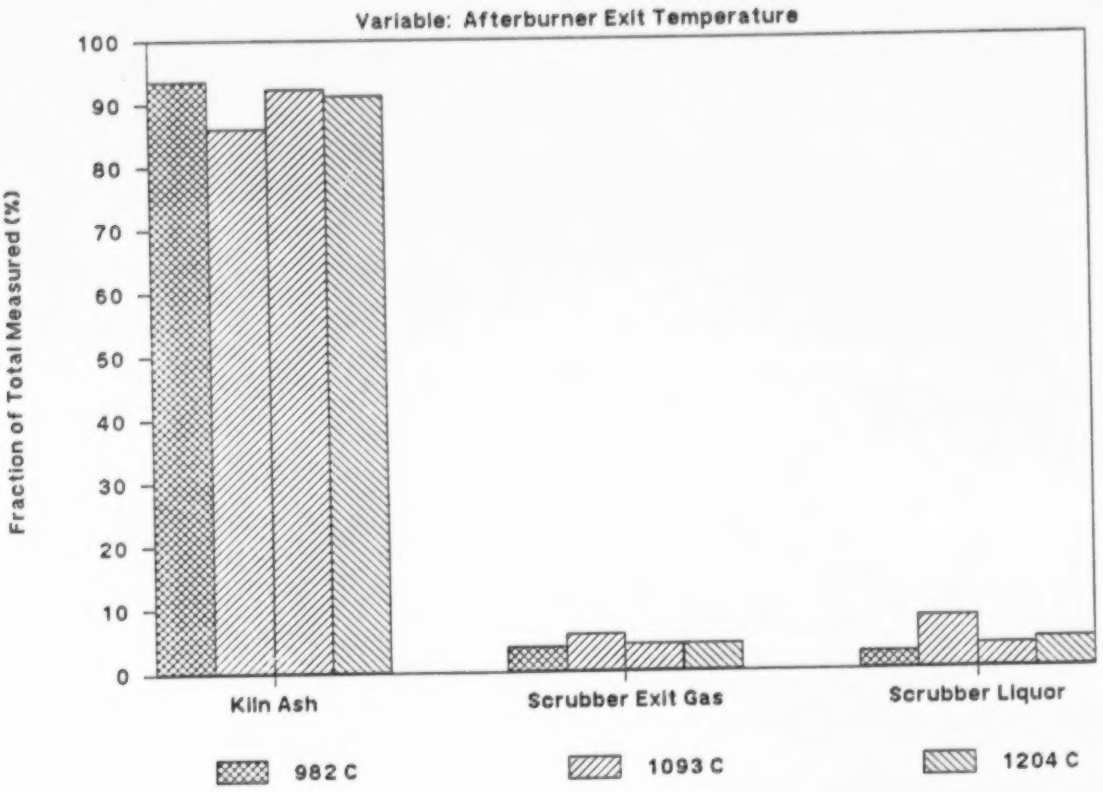


Figure 11b. Arsenic discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

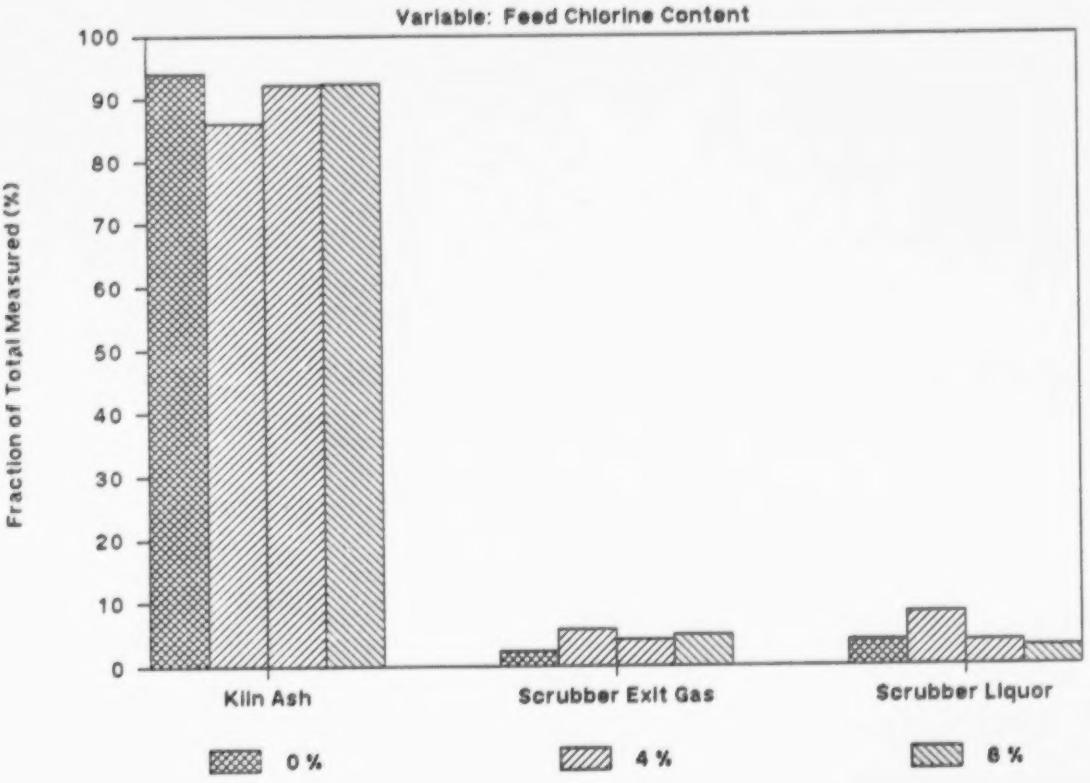


Figure 11c. Arsenic discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

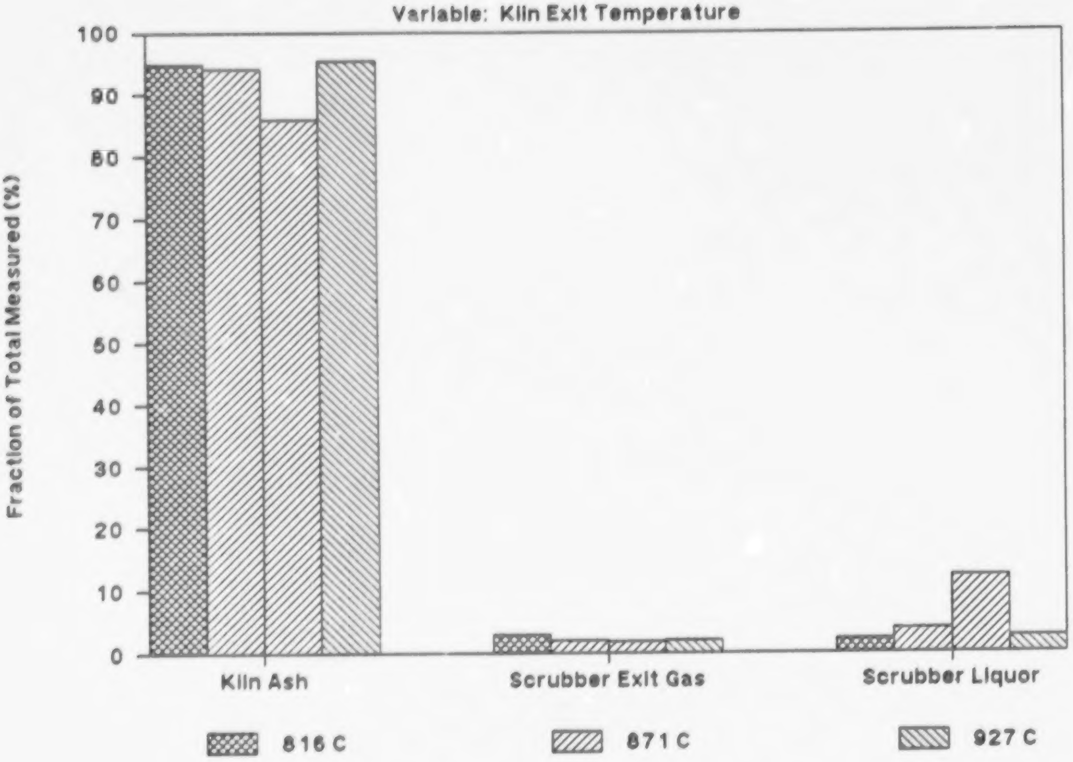


Figure 12a. Chromium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

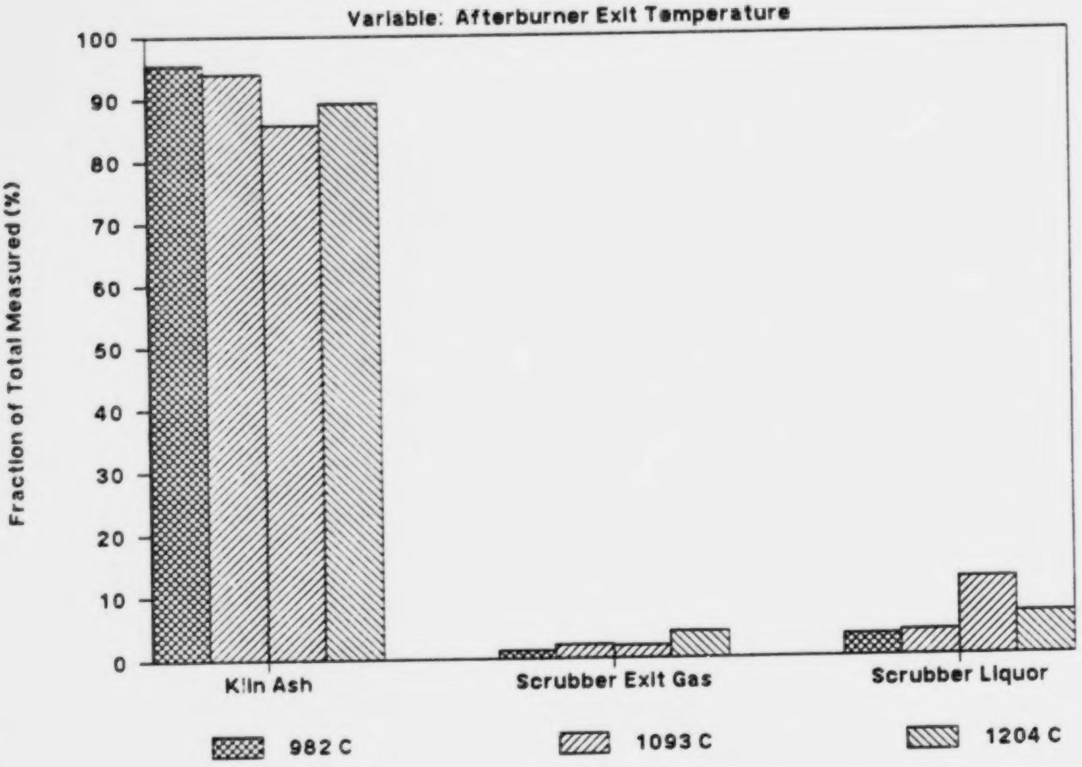


Figure 12b. **Chromium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.**

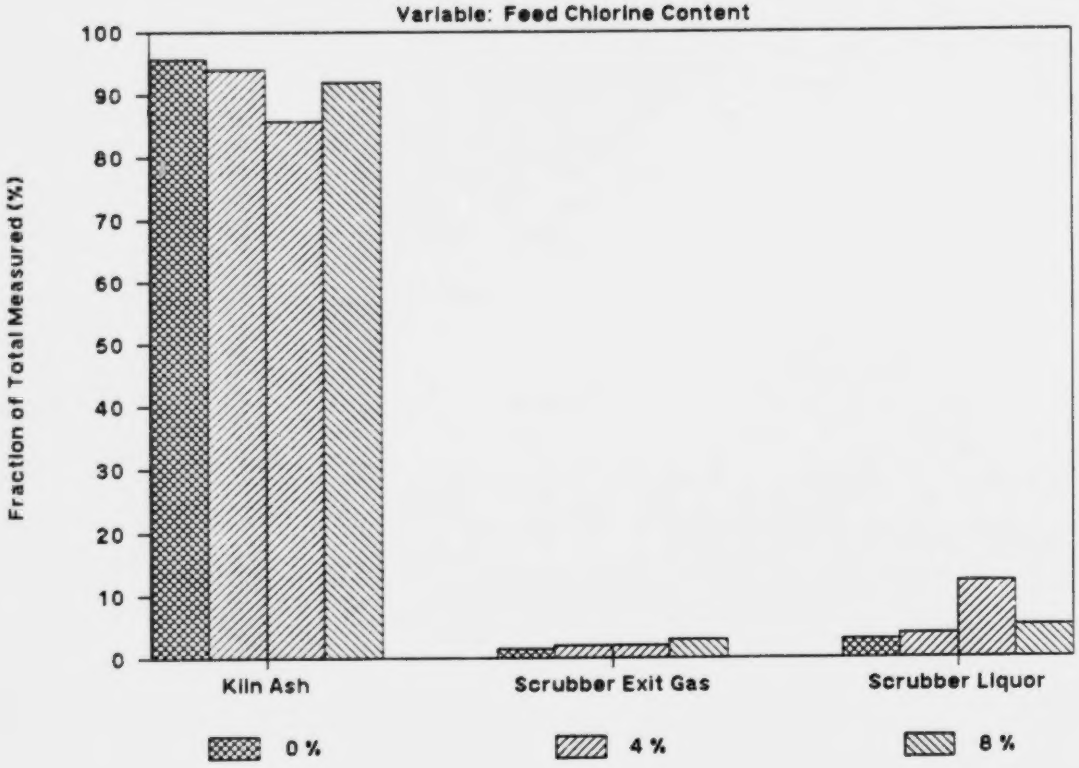


Figure 12c. Chromium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

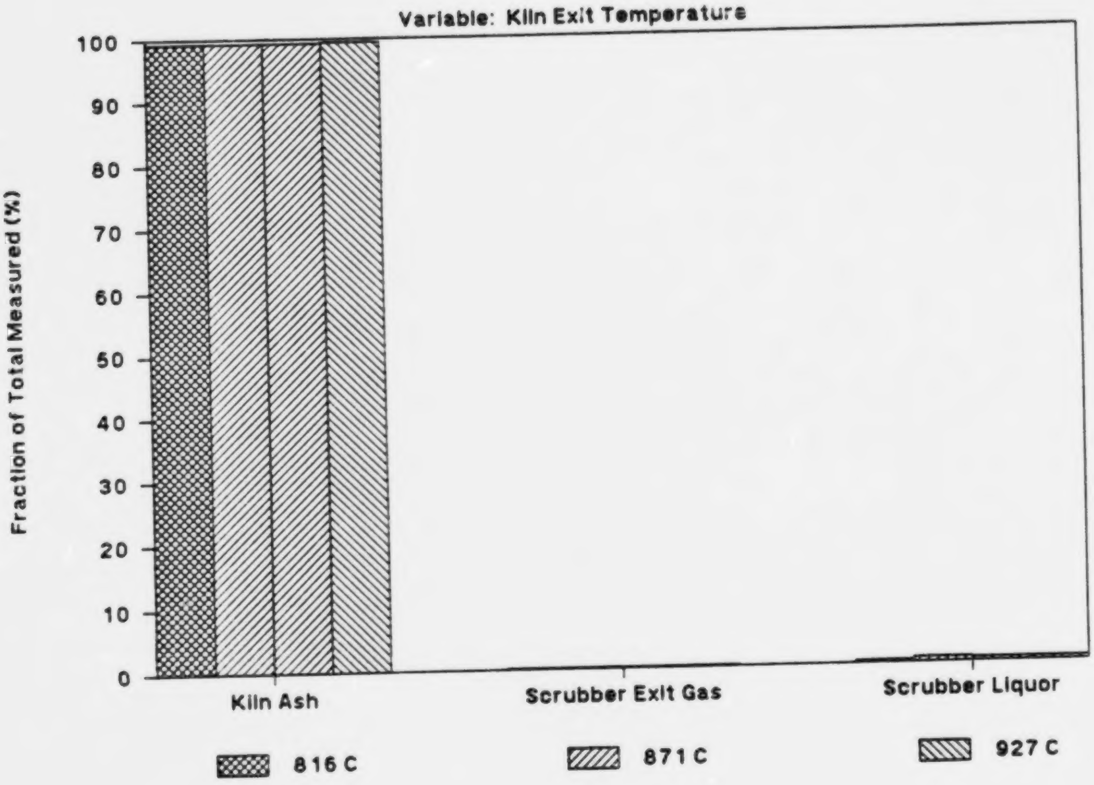


Figure 13a. Magnesium discharge distributions for the parametric trace metal tests: effect of kiln temperature.

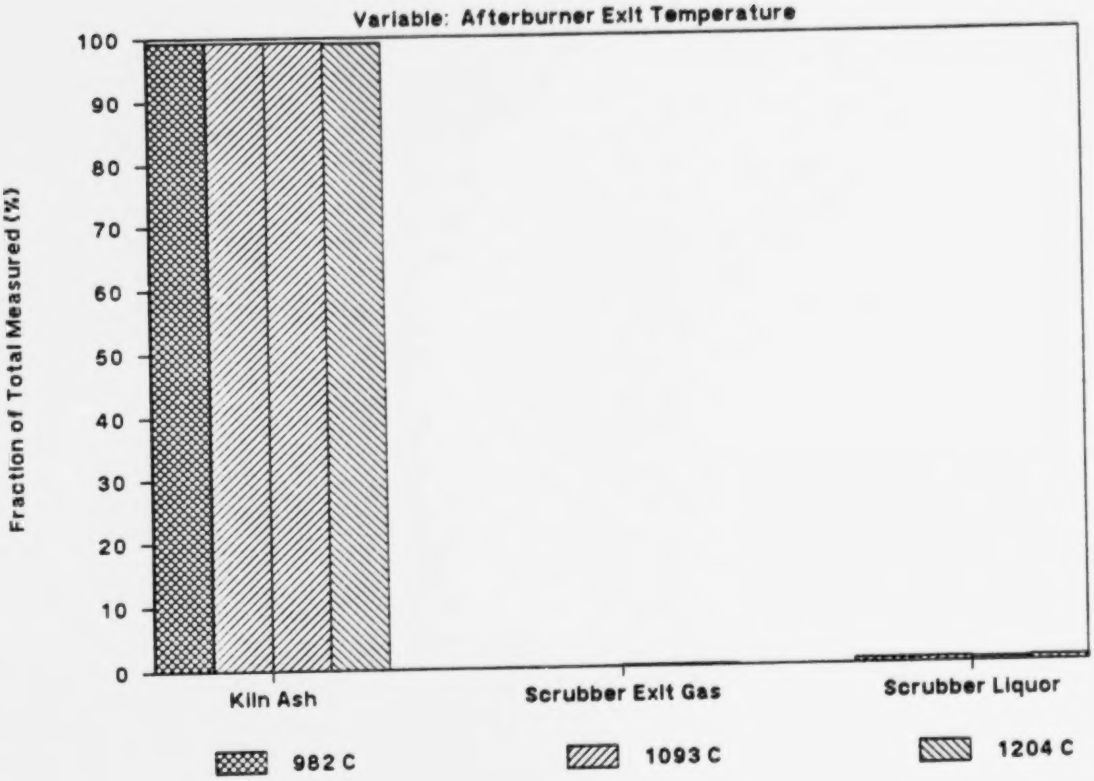


Figure 13b. Magnesium discharge distributions for the parametric trace metal tests: effect of afterburner temperature.

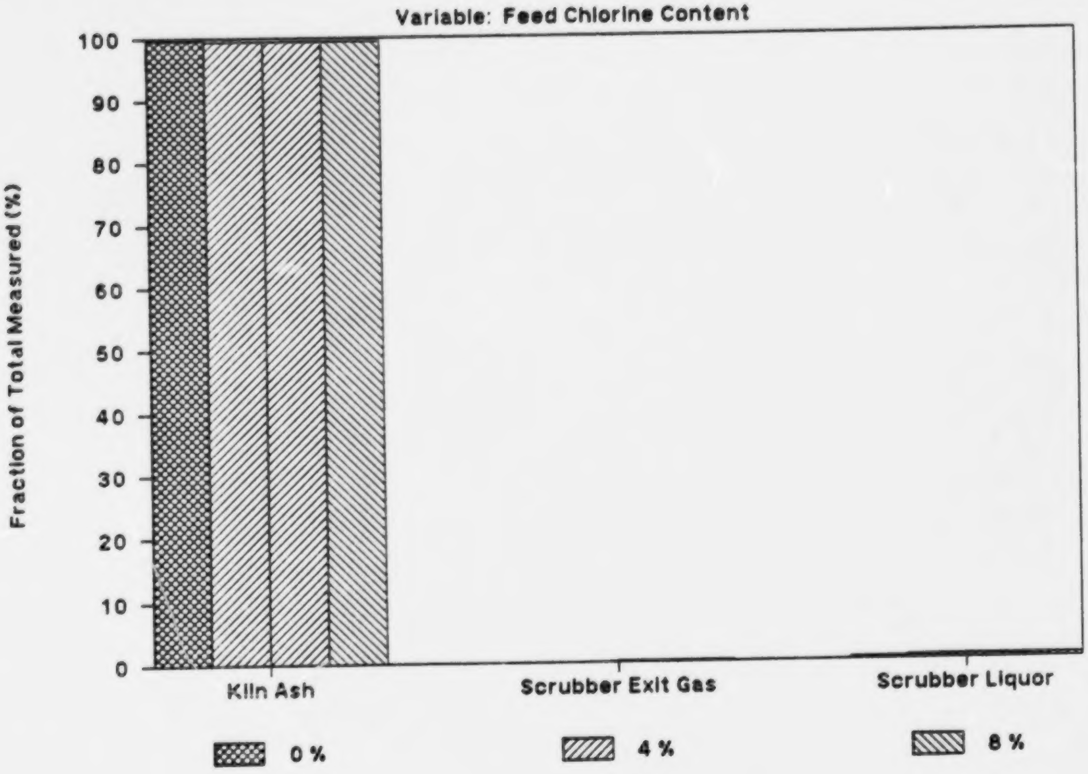


Figure 13c. Magnesium discharge distributions for the parametric trace metal tests: effect of feed chlorine content.

The following discussion focuses on each metal's discharge distribution data in turn.

Cadmium

The data in Figure 5 show that the kiln ash accounted for about 10 percent of the measured discharge cadmium provided there was chlorine in the feed mixture. The data suggest that the kiln ash fraction decreases slightly with increasing kiln temperature. A corresponding increase in the scrubber exit flue gas with a decrease in the scrubber liquor cadmium fraction seems to have occurred with increasing temperature, although the data show some variability. This behavior would be consistent with expectations. Increasing kiln temperature would be conducive to volatilizing more cadmium in the kiln. Any cadmium vaporized in the kiln would remain in the vapor phase through the afterburner, then condense as the flue gas was quenched. In accordance with the volatilization/condensation discussion in Section 4.4.2.1, scrubber efficiency would be expected to decrease. This would give rise to a greater scrubber exit flue gas fraction and a smaller scrubber liquor fraction.

No clear trend in any discharge stream's cadmium fraction, within the apparent data variability, is seen with varying afterburner temperatures.

The kiln ash cadmium fraction is clearly greater when no chlorine is in the feed than when chlorine is present. This would be expected if cadmium chlorides were more volatile than cadmium oxides.

Lead

The data in Figure 6 show that, as for cadmium, the kiln ash accounted for slightly greater than 10 percent of the measured discharge lead, provided there was chlorine in the feed. Again, there appears to be a slight decrease in kiln ash lead fraction with increasing kiln temperature, with a corresponding increase in scrubber exit flue gas fraction and a decrease in scrubber liquor fraction.

Interestingly, there appeared to be a decrease in scrubber exit flue gas lead fraction and an increase in scrubber liquor fraction with increasing afterburner temperature. This seems counter to expectations.

As with cadmium, the kiln ash lead fraction was substantially greater for the test with no feed chlorine than for the tests with 4 percent chlorine in the feed. This is consistent with expectations. The principal vapor phase lead species in a chlorine-free excess oxygen environment is expected to be lead metal with a volatility temperature of 627°C (1160°F). However, in a chlorine-containing environment, the principal vapor phase species becomes $PbCl_4$ with a volatility temperature of -15°C (5°F).

The data in Figure 6 show a further decrease in kiln ash lead fraction when feed chlorine content was further increased from 4 to 8 percent. Scrubber exit flue gas lead fraction monotonically increases with increased feed chlorine content. Scrubber efficiency correspondingly decreases.

Bismuth

The data in Figure 7 show bismuth behaves in a manner similar to cadmium and lead with respect to feed chlorine content. The kiln ash bismuth fraction was in the 20 to 35 percent range for all tests in which the feed contained chlorine. A marked increase in kiln ash bismuth fraction (a decrease in bismuth volatility) occurred when chlorine was removed from the feed. A modest increase in scrubber efficiency for bismuth was also observed when feed chlorine was removed.

The data show no significant variation in any discharge stream bismuth fraction with changes in kiln or afterburner temperature.

Barium

The data in Figure 8 suggest that no test variable had a significant effect on barium discharge distributions.

Copper

The data in Figure 9 show that neither kiln nor afterburner temperatures had measurable effects on copper discharge distributions. However, copper kiln ash fractions clearly decreased monotonically with increasing feed chlorine content. Scrubber exit flue gas copper fractions showed a corresponding monotonic increase with increasing feed chlorine content. Both of these observations would be consistent with expectations if copper chlorides were more volatile than corresponding oxides.

Interestingly, scrubber liquor copper fractions may have increased slightly with increasing feed chlorine content. This suggests that the increased solubility of copper chlorides may have somewhat offset the partitioning of copper to finer particle size with increased copper volatilization, and thereby offset to some degree the expected decrease in scrubber collection efficiency.

Strontium

The data in Figure 10 show that neither kiln temperature nor feed chlorine content affect strontium discharge distributions. There was an apparent steady decrease in kiln ash strontium fraction with increasing afterburner temperature. This is coincidental, since afterburner temperature can have no effect on kiln ash fraction. The apparent steady increase in scrubber liquor strontium fraction is most likely similarly coincidental.

Scrubber exit flue gas strontium fraction appeared to increase slightly with increasing afterburner temperature. Although this may also have been coincidental, it would be expected if higher afterburner temperature caused an increase in the amount of strontium in entrained flyash from the kiln which vaporized in the afterburner.

Arsenic

The data in Figure 11 show no effect of afterburner temperature on arsenic discharge distributions. A slight decrease in kiln ash fraction, accompanied by a slight increase in scrubber exit flue gas fraction, with increasing kiln temperature appears to have occurred. A decrease in scrubber efficiency was observed with increasing feed chlorine content.

Chromium and Magnesium

The data in Figures 12 and 13 show that no test variable measurably affected chromium or magnesium discharge distributions.

Table 38 summarizes the distribution of each metal between the particulate phase and the vapor/dissolved phase in the afterburner and scrubber exit flue gas for each test. The data in Table 38 represent the individual test data comprising the ranges and averages noted in Table 34. Again, distributions affected by samples with nondetectable levels of a metal are represented by ranges. The range boundaries reflect the assumption that the samples in question contained zero and the detection limit of the metal, respectively. The data in Table 38 show no consistent relationship between particulate/vapor-dissolved phase ratios and test variables.

4.4.2.3 Afterburner Exit Particle Size Distributions

Recall from the sampling and analysis protocol discussion in Section 3, that the flue gas at the afterburner exit was sampled with a sampling train designed to collect a large (~1g) sample of particulate so that this particulate could be size fractionated and trace metal concentrations as a function of particle size could be determined. Sufficient afterburner exit flue gas particulate was collected by the sampling train to allow particle size fractionation to be performed for six of the eight tests performed. Size fractionation was not possible for the particulate catch from tests 5 and 10.

Figure 14 shows a log-probability plot of the total particulate particle size distribution data for the six tests for which size fractionation was performed. As indicated, the size distributions for all tests were roughly log normal. The distributions were roughly comparable for the five tests in which the test waste feed contained chlorine. The size distribution of total particulate was shifted to larger particle size for the one test with no feed chlorine. This would be as expected if the presence of chlorine in the feed served to increase the volatility of feed inorganic constituents. In such a case the recondensation of volatilized inorganic would tend to result in a finer particle size distribution for the chlorine containing feed cases.

Particle size fractions were combined to give four size fraction samples for trace metal analysis. These size fractions were nominally <2, 2 to 4, 4 to 10, and >10 μm respectively. From the analyses of these size fractions, size distributions of each of the test trace metals were obtained.

Figure 15 shows the average size distribution for the five hazardous constituent trace metals included in the test waste mixture (arsenic, barium, cadmium, chromium, and lead) and compares these to the total particulate size distribution. The size distributions shown in Figure 15 represent averages over the six tests for which size distribution analyses were performed. Figure 16 shows analogous size distributions for the four non-hazardous trace metals included in the test waste mixture (bismuth, copper, magnesium, and strontium).

The data in Figures 15 and 16 show that the average size distributions for chromium, copper, magnesium, and strontium were coarser (shifted to larger particle sizes) than the overall particulate size distribution. These are all relatively nonvolatile metals with high volatility temperatures (See Tables 31 and 32). The average size distributions for barium and bismuth were finer (shifted to smaller particle sizes) than the overall particulate. These two are more volatile, having lower volatility temperature than chromium, copper, magnesium, and strontium.

Figures 17 through 22 show individual test metal size distributions for both the hazardous constituent and non-hazardous constituent trace metals.

TABLE 38. PHASE DISTRIBUTION OF FLUE GAS METALS IN THE AFTERBURNER AND SCRUBBER EXIT FLUE GAS FOR THE TRACE METALS PARAMETRIC TEST SERIES

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
						%						
Arsenic												
Afterburner exit:												
Particulate	–	91	–	91	>97	91	–	94	47	91	–	65
Vapor/dissolved phase	–	9	–	9	<3	9	–	6	53	9	–	35
Scrubber exit:												
Particulate	>76	>72	>53	81-95	>74	>72	>53	>74	32	>72	>53	>78
Vapor/dissolved phase	<24	<28	<47	5-19	<26	<28	<47	<26	68	<28	<47	<22
Barium												
Afterburner exit:												
Particulate	–	3	–	1	3	3	–	1	6-10	3	–	3
Vapor/dissolved phase	–	97	–	99	97	97	–	99	90-94	97	–	97
Scrubber exit:												
Particulate	55	26-29	10-27	31-33	25-29	26-29	10-27	32-35	25	26-29	10-27	27
Vapor/dissolved phase	45	71-74	73-90	67-69	71-75	71-74	73-90	65-68	75	71-74	73-90	73
Bismuth												
Afterburner exit:												
Particulate	–	17	–	31	37	17	–	12-20	23	17	–	9
Vapor/dissolved phase	–	83	–	69	63	83	–	80-88	77	83	–	91
Scrubber exit:												
Particulate	90-94	83	70-76	86-93	73	83	70-76	86	>75	83	70-76	93-99
Vapor/dissolved phase	6-10	17	24-30	7-14	27	17	24-30	14	<25	17	24-30	1-7

(continued)

TABLE 38. (continued)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
						%						
Cadmium												
Afterburner exit:												
Particulate	–	57	–	92	73	57	–	82	68	57	–	82
Vapor/dissolved phase	–	43	–	8	27	43	–	18	32	43	–	18
Scrubber exit:												
Particulate	84-89	>94	>86	87	95-99	>94	>86	>94	>82	>94	>86	>95
Vapor/dissolved phase	10-16	<6	<14	13	1-5	<6	<14	<6	<18	<6	<14	<5
Chromium												
Afterburner exit:												
Particulate	–	–	–	–	–	–	–	–	–	–	–	–
Vapor/dissolved phase	–	–	–	–	–	–	–	–	–	–	–	–
Scrubber exit:												
Particulate	79-84	>82	68-90	81-92	70	>82	68-90	87	77-94	>82	68-90	>85
Vapor/dissolved phase	16-21	<18	10-32	8-19	30	<18	10-32	13	6-23	<18	10-32	<15
Copper												
Afterburner exit:												
Particulate	–	58	–	80	55	58	–	77	29	58	–	72
Vapor/dissolved phase	–	42	–	20	45	42	–	23	71	42	–	28
Scrubber exit:												
Particulate	97	97	45	87	80	97	45	89	51-57	97	45	99
Vapor/dissolved phase	3	3	55	13	20	3	55	11	43-49	3	55	1

(continued)

TABLE 38. (concluded)

Test:	5	7	10	6	9	7	10	8	4	7	10	11
Primary variable:	Kiln exit temperature (°C)				Afterburner exit temperature (°C)				Feed chlorine content (wt %)			
Target:	816	871	871	927	982	1093	1093	1204	0	4	4	8
Test average:	825	878	873	928	983	1088	1094	1196	0.0	3.8	4.6	8.3
Held constant:	AB exit = 1093°C; chlorine = 4%				Kiln exit = 871°C; chlorine = 4%				Kiln exit = 871°C; AB exit = 1093°C			
Test average	1071	1088	1094	1092	875	878	873	871	874	878	873	870
Test average:	3.7	3.8	4.6	4.2	3.4	3.8	4.6	3.6	1093	1088	1094	1092
						%						
Lead												
Afterburner exit:												
Particulate	–	28	–	76	54	28	–	66	26	28	–	71
Vapor/dissolved phase	–	72	–	24	46	72	–	34	74	72	–	29
Scrubber exit:												
Particulate	90	>99	93	90	>99	>99	93	>99	>95	>99	93	>99
Vapor/dissolved phase	10	<1	7	10	<1	<1	7	<1	<5	<1	7	<1
Magnesium												
Afterburner exit:												
Particulate	–	69	–	70	91	69	–	84	57	69	–	76
Vapor/dissolved phase	–	31	–	30	9	31	–	16	43	31	–	24
Scrubber exit:												
Particulate	93	73	25	82-88	62	73	25	71	65	73	25	72
Vapor/dissolved phase	7	27	75	12-18	38	27	75	29	35	27	75	28
Strontium												
Afterburner exit:												
Particulate	–	41	–	41	58	41	–	19-25	8-34	41	–	25-32
Vapor/dissolved phase	–	59	–	59	42	59	–	75-81	66-92	59	–	68-75
Scrubber exit:												
Particulate	32	79-85	47	89-98	>91	79-85	47	85-89	74-80	79-85	47	76-86
Vapor/dissolved phase	68	15-21	53	2-11	<9	15-21	53	11-15	20-26	15-21	53	14-24

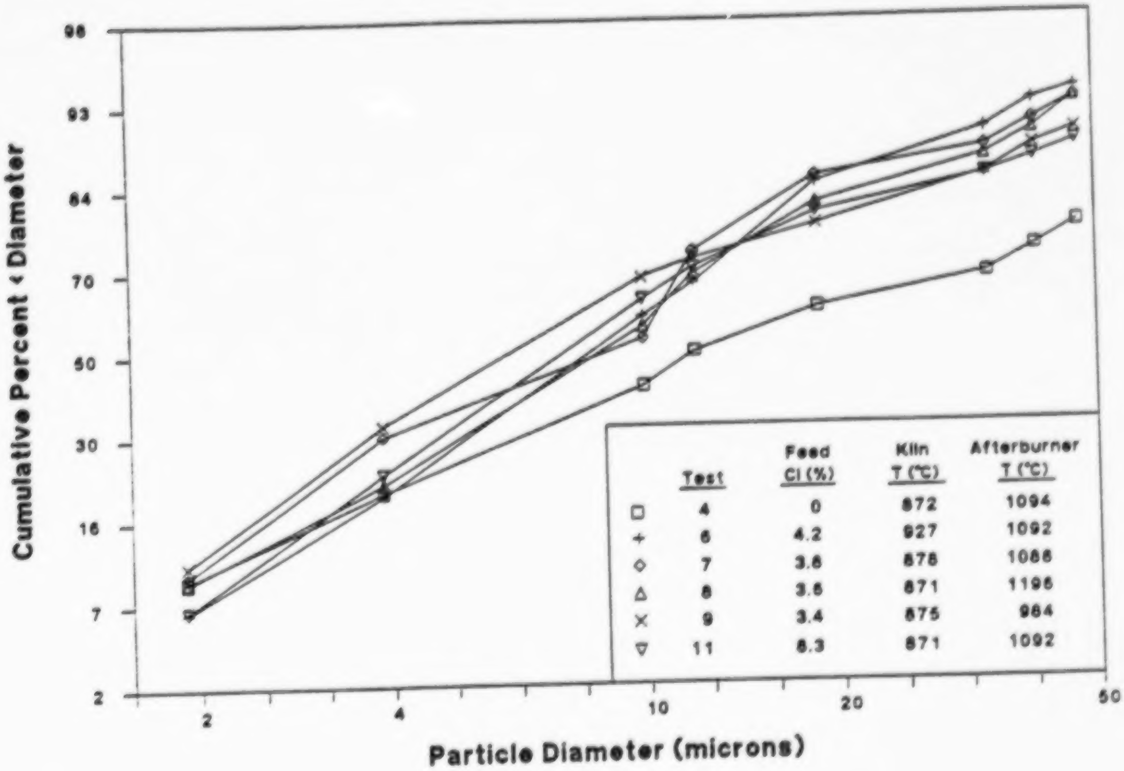


Figure 14. Afterburner exit flue gas particulate size distributions for the parametric trace metal tests.

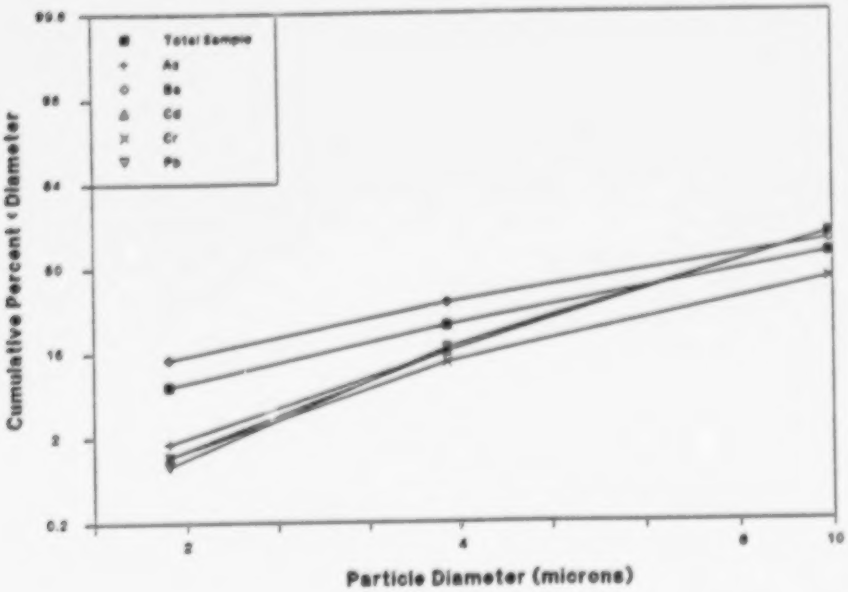


Figure 15. Hazardous constituent trace metal size distributions in afterburner exit flue gas particulate for the parametric trace metal tests.

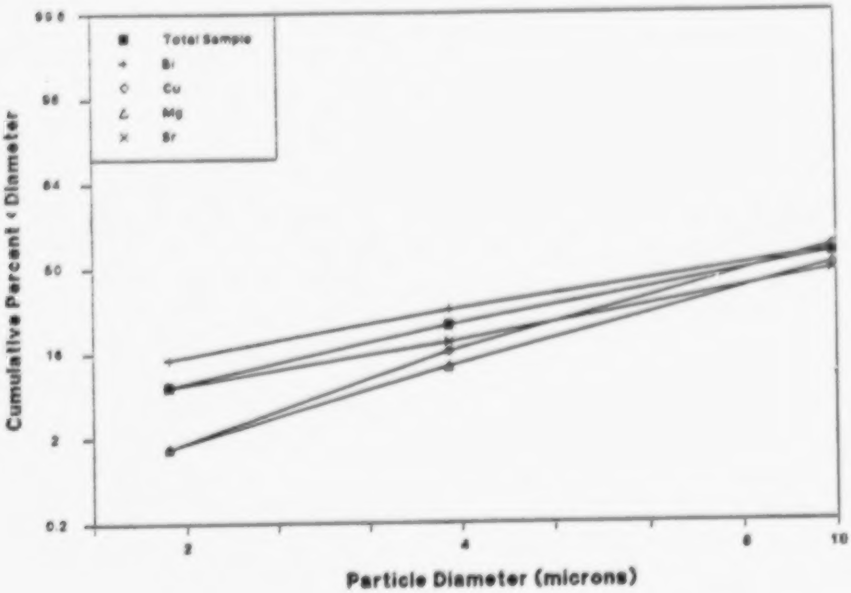


Figure 16. Nonhazardous trace metal size distributions in afterburner exit flue gas particulate for the parametric trace metal tests.

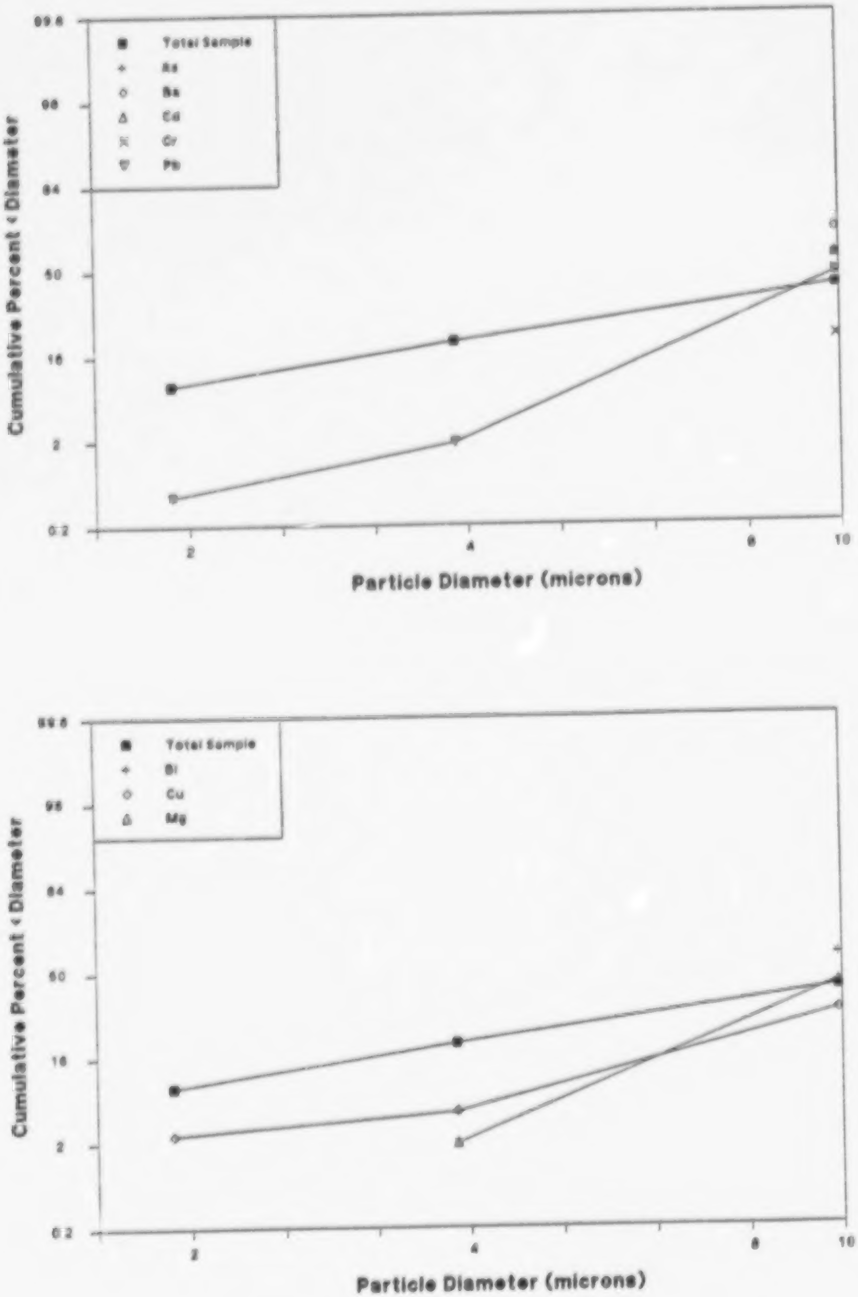


Figure 17. Trace metal size distributions in afterburner exit flue gas for Test 4.

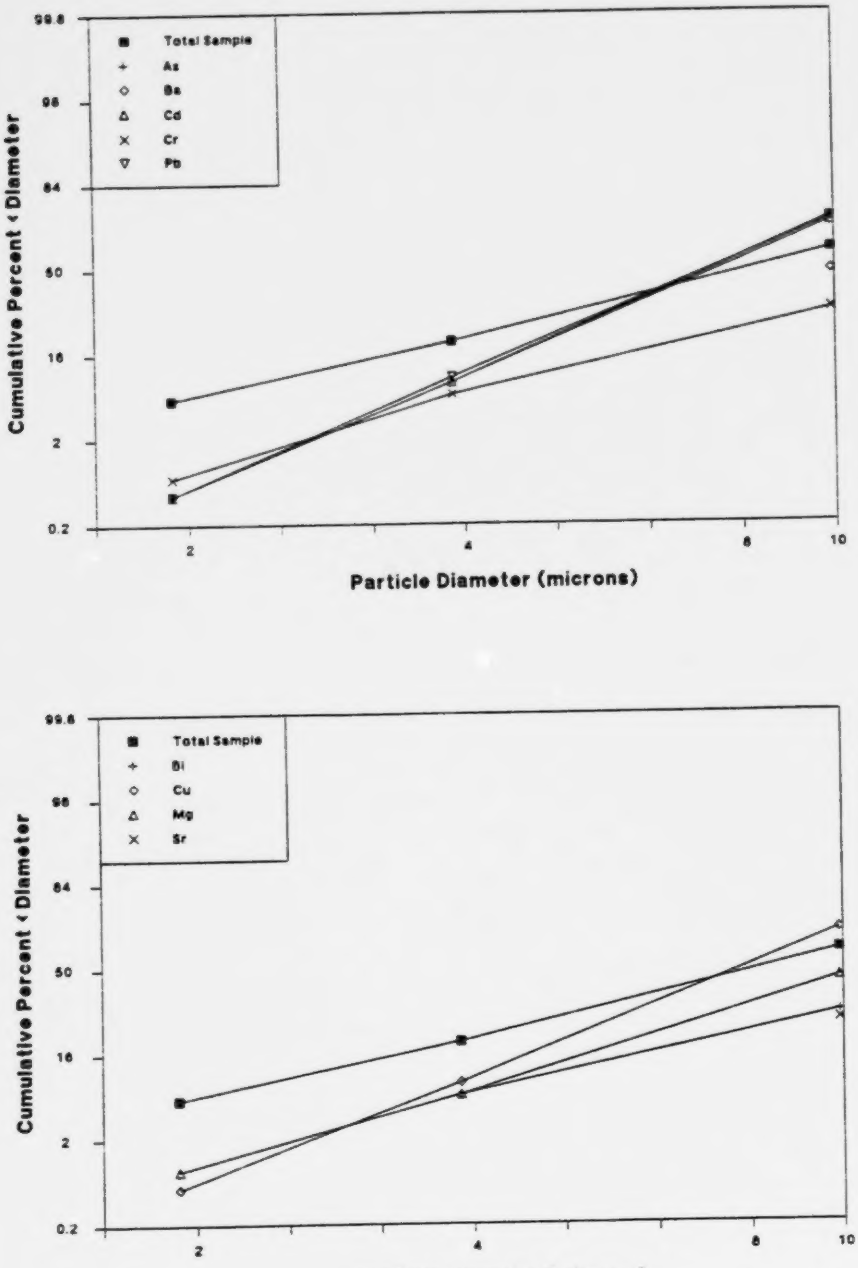
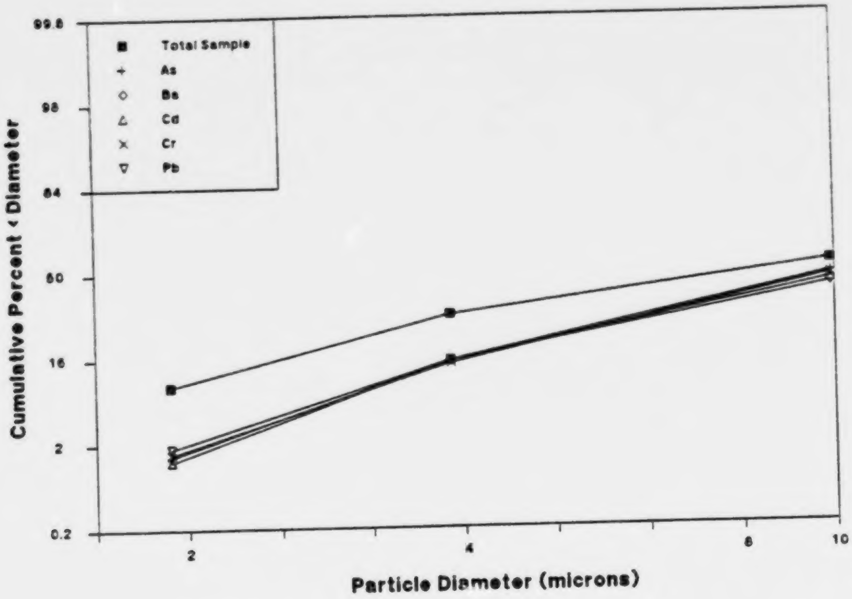


Figure 18. Trace metal size distributions in afterburner exit flue gas for Test 6.



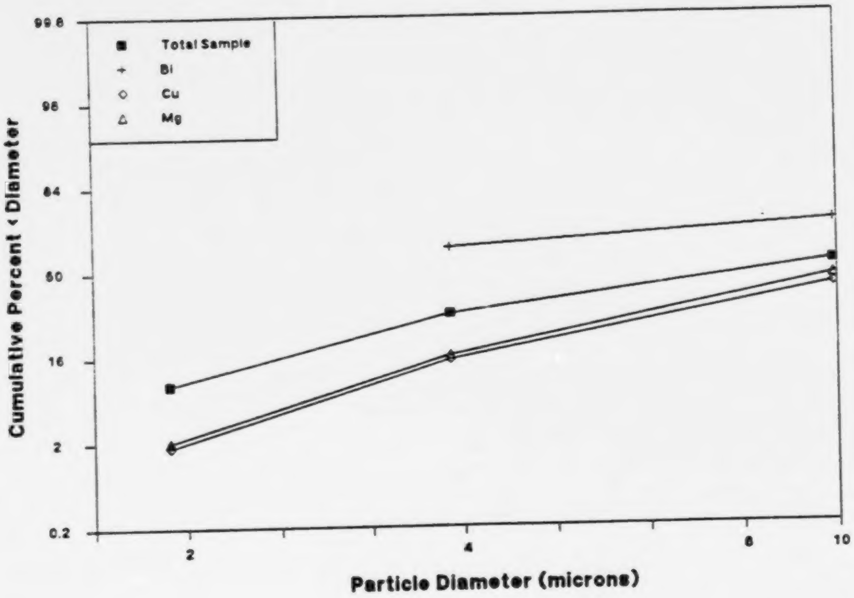
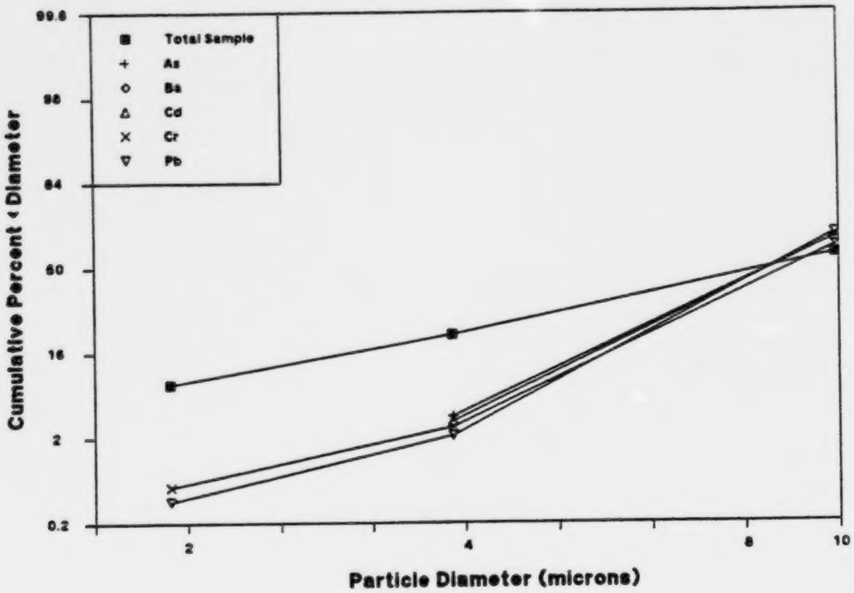


Figure 19. Trace metal size distributions in afterburner exit flue gas for Test 7.



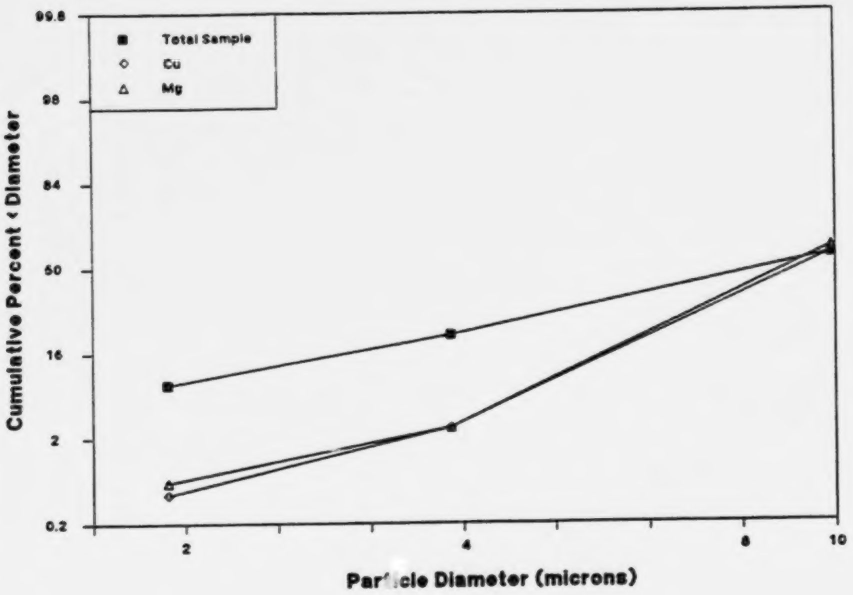
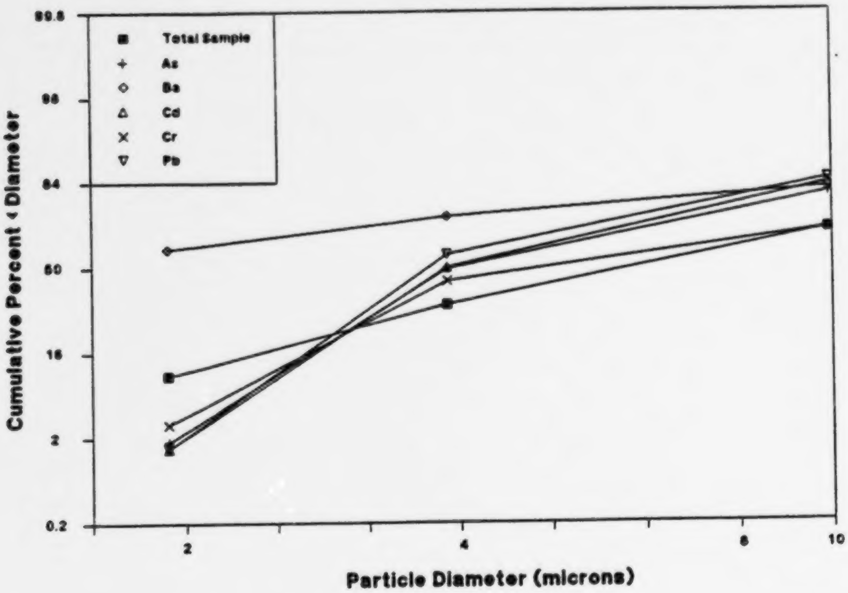


Figure 20. Trace metal size distributions in afterburner exit flue gas for Test 8.



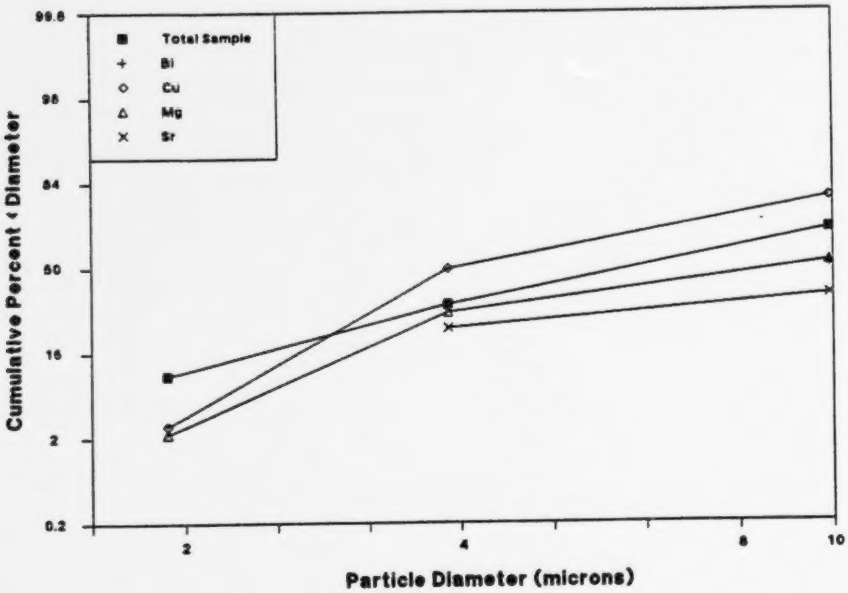


Figure 21. Trace metal size distributions in afterburner exit flue gas for Test 9.

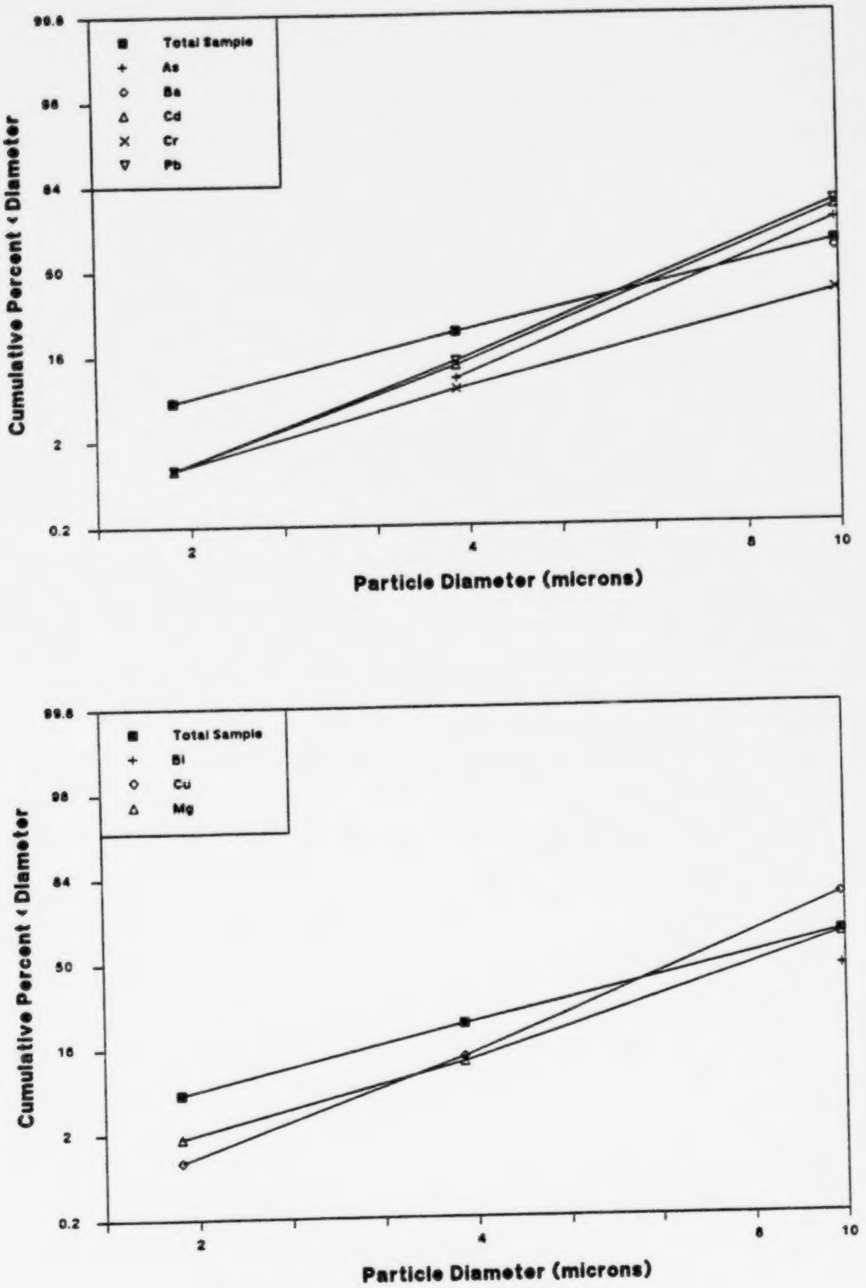


Figure 22. Trace metal size distributions in afterburner exit flue gas for Test 11.

4.5 POHC DESTRUCTION AND REMOVAL EFFICIENCIES

The three compounds introduced in the synthetic waste feed as principal organic hazardous constituents (POHCs) for these tests were toluene, chlorobenzene, and tetrachloroethylene. Flue gas concentrations of the compounds measured at the three locations sampled (the afterburner exit, the scrubber system exit, and the stack (three tests)) during the parametric trace metal tests are summarized in Table 39. The data in the table show that POHC concentrations were generally decreased in the scrubber exit flue gas compared to the afterburner exit flue gas; although toluene and chlorobenzene concentrations increased for Tests 5, 6, and 9. Reduced concentrations are expected due to some combination of flue gas dilution via air inleakage and POHC removal in the scrubber system. The increased concentrations observed for Tests 5, 6, and 9 are possibly related to a sampling artifact in either the afterburner or scrubber exit sampling trains.

Table 40 combines the flue gas concentration data from Table 39 with the flue gas flowrate data in the sampling sheets in Appendix C, the synthetic liquid waste feed composition data from Table 17, and the liquid waste feedrate data from Table 4, to give the POHC DREs at the three locations sampled. As shown in the table, POHC DREs in the stack ranged from 99.9975 to 99.9994 percent. With the exception of the afterburner exit for Test 11, POHC DREs were above 99.99 percent at all locations for all tests. For Test 11 with 8.4 percent waste chlorine feed, the POHC DREs were uniformly lower than the other tests (with lower feed chlorine contents) at all three sampling locations.

4.6 VOLATILE PRODUCTS OF INCOMPLETE COMBUSTION (PICs)

As noted in Section 3, all VOST traps were analyzed by thermal desorption, purge and trap, GC/FID for the volatile organic constituents listed in Table 16. This list includes the POHCs discussed in Section 4.5. However, several other potential volatile PICs are included in this analyte list as well. Table 41 summarizes the flue gas concentrations of the non-POHC volatile organic constituents detected in the afterburner exit flue gas for one or more of the tests performed. Tabulated values represent the average of the three VOST trap pairs operated during each test. For determining the average emission rate, practical quantitation limits (PQLs) were used for trains containing concentrations less than the PQL. Table 42 presents an analogous summary for the scrubber system exit flue gas.

The data in Table 41 show that the major PICs detected in the afterburner exit flue gas were chloroform, carbon tetrachloride, and benzene. Concentrations ranged from about 1 to 12 μg/dscm. Note that for Test 4 (0 percent chlorine feed), the chlorinated PICs are present at significantly reduced concentrations, as expected. Also for Test 4, both tetrachloroethylene and chlorobenzene were detected, and are reported as PICs since neither was a POHC for this test.

Table 42 shows that chloroform levels in the scrubber exit flue gas were above those at the afterburner exit for five of the eight tests. The origin of the increased chloroform is likely the scrubber makeup water. The scrubber water source is a chlorine treated potable supply in which the trihalomethane (THM) compounds are typically found. These compounds volatilize readily; the scrubber essentially acts as a purger, releasing the THM into the flue gas stream with the entrained water vapor.

For all tests, carbon tetrachloride levels in the scrubber exit flue gas were less than the levels in the afterburner exit flue gas. Benzene concentrations increased in the four tests, decreased in three tests, and remained the same in the one test. No correlations between PIC levels in the flue gas at either location and corresponding POHC DRE are apparent.

TABLE 39. FLUE GAS POHC CONCENTRATIONS FOR THE PARAMETRIC TRACE METAL TESTS

		POHC concentration (μg/dscm)								
		Afterburner exit			Scrubber exit			Stack		
Test	Test date	Toluene	Chlorobenzene	Tetrachloroethylene	Toluene	Chlorobenzene	Tetrachloroethylene	Toluene	Chlorobenzene	Tetrachloroethylene
4	9/14/88	85	—	—	48	—	—	—	—	—
5	8/25/88	10	2.1	7.5	40	3.3	5.6	—	—	—
6	9/16/88	14	1.8	13	73	7.4	10	—	—	—
7	8/30/88	140	17	35	21	2.4	3.1	—	—	—
8	9/07/88	240	29	43	40	4.2	5.1	99	13	14
9	9/09/88	19	2.2	5.0	27	3.3	4.2	85	14	7.9
10	9/20/88	510	86	55	21	1.9	4.0	—	—	—
11	9/22/88	1300	550	750	62	18	24	120	22	29

TABLE 40. POHC DREs FOR THE PARAMETRIC TRACE METAL TESTS

		DRE (%)								
		Afterburner exit			Scrubber exit			Stack		
Test	Test date	Toluene	Chlorobenzene	Tetrachloroethylene	Toluene	Chlorobenzene	Tetrachloroethylene	Toluene	Chlorobenzene	Tetrachloroethylene
4	9/14/88	99.99943	—	—	99.99936	—	—	—	—	—
5	8/25/88	99.999910	99.999913	99.99966	99.99930	99.99973	99.99945	—	—	—
6	9/16/88	99.999903	99.999923	99.99948	99.9988	99.99932	99.99907	—	—	—
7	8/30/88	99.9990	99.99919	99.9984	99.99967	99.99976	99.99969	—	—	—
8	9/07/88	99.9980	99.9986	99.9980	99.99915	99.99949	99.99940	99.9986	99.9990	99.9990
9	9/09/88	99.99982	99.99988	99.99974	99.99951	99.99966	99.99957	99.9988	99.9990	99.9994
10	9/20/88	99.9967	99.9967	99.9979	99.99967	99.99987	99.99964	—	—	—
11	9/22/88	99.988	99.989	99.985	99.9986	99.99918	99.9989	99.9975	99.9990	99.9987

TABLE 41. AFTERBURNER EXIT FLUE GAS VOLATILE PIC CONCENTRATIONS

Test Test date	4 (9-14-88)	5 (8-25-88)	6 (9-16-88)	7 (8-30-88)	8 (9-07-88)	9 (9-09-88)	10 (9-20-88)	11 (9-22-88)
Compound	Afterburner exit flue gas concentration (µg/dscm)							
Chloroform	2.4	9.1	5.3	12.6	5.4	9.0	6.4	4.9
Carbon tetrachloride	1.6	8.4	5.3	3.1	5.5	5.5	11.9	<1.5
Benzene	7.8	2.7	<1.6	2.2	9.9	10.6	7.5	11.7
Trichloroethylene	<1.7	1.7	<1.7	<1.7	<1.7	1.9	1.9	<1.7
1,3-Dichlorobenzene	<1.6	2.0	<1.6	<1.6	<1.6	<1.6	<1.6	<1.6
1,4-Dichlorobenzene	<1.6	2.1	<1.6	1.7	1.6	1.6	<1.6	<1.6
1,2-Dichlorobenzene	1.9	<1.5	<1.5	<1.5	1.7	<1.5	2.2	<1.5
Tetrachlorethylene	16.8	P[a]	P	P	P	P	P	P
Chlorobenzene	2.0	P	P	P	P	P	P	P

[a] P: Compound was a POHC for these tests.

TABLE 42. SCRUBBER EXIT FLUE GAS VOLATILE PIC CONCENTRATIONS

Test Test date	4 (9-14-88)	5 (8-25-88)	6 (9-16-88)	7 (8-30-88)	8 (9-07-88)	9 (9-09-88)	10 (9-20-88)	11 (9-22-88)
Compound	Scrubber exit flue gas concentration (µg/dscm)							
Chloroform	4.3	10.0	8.4	3.3	14.7	5.6	17.9	27.7
Carbon tetrachloride	<1.5	2.1	2.1	1.9	4.7	2.7	3.6	2.3
Benzene	<1.6	8.5	4.8	<1.6	10.0	16.7	15.7	5.8
1,3-Dichlorobenzene	<1.6	1.7	1.6	<1.6	1.9	1.9	<1.6	<1.6
1,4-Dichlorobenzene	<1.6	1.6	1.5	<1.6	1.8	2.2	2.5	<1.6
1,2-Dichlorobenzene	2.3	<1.5	3.0	<1.5	<1.5	2.4	4.6	1.6
Tetrachlorethane	<1.8	<1.8	<1.8	<1.8	7.8	14.1	<1.8	<1.8
1,2-Dichloroethane	<1.5	<1.5	<1.5	2.1	<1.5	<1.5	2.3	<1.5
Ethylbenzene	6.4	6.3	6.0	3.2	<1.5	10.0	7.5	3.1

SECTION 5

CONCLUSIONS

A five-week series of pilot-scale incineration tests was performed to evaluate the fate of trace metals fed to a rotary kiln incinerator equipped with a venturi scrubber/packed column scrubber for particulate/acid gas control. Three tests focused on determining the valence state (trivalent/hexavalent) of chromium in emissions and discharges from the incinerator, and investigating whether valence state distributions were affected by the chromium valence state in the waste feed to the incinerator or the feed chlorine content. Eight tests focused on a parametric evaluation of the fate of five hazardous constituent trace metals (arsenic, barium, cadmium, chromium, and lead), and four nonhazardous constituent trace metals (bismuth, copper, magnesium, and strontium) fed to the incinerator. Test variables for the parametric evaluation were kiln temperature, afterburner temperature, and feed chlorine content. A factorial experimental matrix was tested in which kiln temperature was varied from 816 to 927°C (1500 to 1700°F), afterburner temperature was varied from 982 to 1204°C (1800 to 2200°F), and feed chlorine content was varied from 0 to 8 percent.

For all tests, the waste feed consisted of a synthetic mixture prepared by combining an organic liquid mixture with a clay absorbent material. The organic liquid mixture consisted of toluene with varying amounts of tetrachloroethylene and chlorobenzene added to give desired feed chlorine content. This synthetic solid waste material contained nominally 0.4 kg of organic liquid to 1 kg of clay absorbent. This mixture was introduced to the kiln via a screw feeder. Test trace metals were prepared in an aqueous solution. This solution was metered into the clay/organic liquid mixture in the screw feeder.

For the chromium valence state tests, total chromium and hexavalent chromium (Cr(+6)) concentrations were measured in the kiln ash and scrubber blowdown discharges as well as in the incinerator flue gas at two locations: the afterburner exit and downstream of the scrubber system. For the parametric trace metal tests, trace metal concentrations were measured in the same emission/discharge streams. In addition, the distribution of flue gas trace metal between particulate and vapor/dissolved phases was measured, and the particulate size distribution of trace metals in the afterburner exit flue gas was determined.

Conclusions from the chromium valence state tests include:

- Most of the chromium fed was discharged in the kiln ash. Kiln ash accounted for 95 percent of the discharge amount with no chlorine in the feed, regardless of the chromium valence state in the feed. The kiln ash fraction decreased to 84 percent when chlorine was present in the feed.

- Scrubber exit flue gas accounted for 1 to 2 percent of the chromium discharged with no chlorine in the feed, again regardless of the feed valence state. Scrubber exit flue gas fraction increased to 4 percent when chlorine was present in the feed.

- Scrubber liquor accounted for 3 percent of the discharged chromium with no feed chlorine. The scrubber liquor fraction increased to 11 percent with chlorine-containing feed.

- The kiln ash contained negligible Cr(+6) regardless of feed chromium valence state or chlorine content.

- Nominally 15 percent of the chromium in the scrubber exit flue gas was Cr(+6) with no feed chlorine. The Cr(+6) fraction increased to almost 50 percent of the scrubber exit flue gas chromium with chlorine present in the feed. This result is consistent with the formation of chromyl chloride from entrained chromium vaporized in the afterburner.

- Scrubber liquor chromium was 20 to 30 percent Cr(+6) with Cr(+3) feed; scrubber liquor chromium increased to about 60 percent Cr(+6) with Cr(+6) in the feed.

Conclusions from the parametric trace metal tests include:

- Based on normalized discharge distribution data, cadmium, lead, and bismuth were relatively volatile metals with an average of less than about 30 percent of the discharged metal being present in kiln ash. Barium, copper, strontium, arsenic, chromium, and magnesium were relatively nonvolatile with an average of greater than 75 percent of the discharged metal being present in the kiln ash.

- Observed metal volatilities from most to least volatile were cadmium, lead, bismuth, barium, copper, strontium, arsenic, chromium, and magnesium. This order agrees with the order predicted by metal volatility temperature (temperature at which the vapor pressure of a principal vapor species is 10^{-6} atm) with the notable exception of arsenic. Arsenic has the lowest volatility temperature of the metals tested, but was observed to be one of the least volatile of the metals. This suggests that As_2O_3 was not a predominant arsenic species in the incinerator, or that the arsenic is adsorbed by the clay/ash matrix.

- The average apparent scrubber collection efficiency was lower for the volatile metals (36 to 45 percent) than for the nonvolatile metals (49 to 88 percent) with the notable exception of copper. Copper's average scrubber removal efficiency, at about 30 percent, was significantly lower than the efficiencies seen for the other relatively nonvolatile metals.

- Feed chlorine content had a major effect on the partitioning of the volatile metals (cadmium, lead, and bismuth), and of copper. The fraction of metal discharged in the kiln ash for these metals measurably decreased with increasing feed chlorine content.

- Kiln temperature had a minor effect on the relative volatility of the observed most volatile metals (cadmium and lead) and of arsenic. The fraction of metal discharged in the kiln ash decreased slightly with increasing kiln temperature.

- No test variable measurably affected the discharge distributions of barium, strontium, chromium, and magnesium, among the least volatile metals.

- Total afterburner exit flue gas particulate size distributions were roughly lognormal for all tests in which they were measured. The size distributions for all tests with chlorine-containing feed were roughly comparable; the size distribution was shifted to larger particle size with no chlorine in the feed.

- The afterburner exit flue gas average size distributions of the less volatile metals (chromium, copper, magnesium, and strontium) were coarser than the corresponding overall particulate average size distribution. The size distribution for the relatively more volatile metals (barium and bismuth) were finer than the overall particulate average size distribution.

Test waste POHC destruction and removal efficiencies (DREs) were greater than 99.99 percent as measured in the scrubber discharge flue gas and in the stack downstream of the system's carbon bed/HEPA filter for all of the parametric trace metal tests. POHC DREs were similarly greater than 99.99 percent as measured in the afterburner exit flue gas for all tests except the test with the highest (nominally 8 percent) feed chlorine content. In fact, POHC DREs at all locations were uniformly lower for this high-chlorine-content feed than for other tests with lower feed chlorine contents.

Chloroform, carbon tetrachloride, and benzene were the volatile products of incomplete combustion (PICs) present in flue gas at the highest levels, up to the order of 10 μg/dscm.

All test program data quality objectives (DQOs) were met, with the exception of field matrix spike recovery and the precision of feed clay matrix spike duplicate analyses for Cr(+6). Precision and accuracy DQOs for laboratory matrix spikes and field spikes for other matrices were met. The most important Cr(+6) analyses needed to support the test conclusions noted above were for matrices other than the feed clay. Thus, the failure to achieve the recovery and precision DQOs for a single analyte in a single field matrix has very little impact on the test conclusions stated above.

SECTION 6

QUALITY ASSURANCE

A number of quality assurance (QA) procedures were followed to assess the data quality of the laboratory analytical measurements performed in these tests. In addition, QA efforts performed to ensure that data quality is known for the particulate and CEM measurements involved adherence to Reference Method procedures and CEM manufacturers' specifications. No deviations from the quality assurance project plan (QAPP) occurred for these measurements with the exception that the CO/CO_2 monitor at the stack did not function properly. The major laboratory QA efforts focused on the following measurements:

- Volatile organic compounds in the feed mixtures
- Volatile organic compounds in the VOST traps
- Metals in the feed and incinerator discharges

The data quality objectives (DQOs) for these measurements are listed in Table 43. The QA procedures performed included:

- Analyzing replicate feed mixture samples for toluene, chlorobenzene, and tetrachloroethylene
- Preparing a clay/organic liquid feed mixture matrix spike and analyzing replicate samples for spike recovery
- Spiking all VOST train resin traps with two method surrogates and measuring surrogate recovery
- Preparing eight matrix spike VOST traps and analyzing these for spike recovery (seven analyzed at the IRF using the method described in Appendix D and one shipped to the Acurex analytical chemistry laboratory in Mountain View, California and analyzed via EPA Method 5040)
- Obtaining duplicate VOST samples for analysis at both the IRF and the Acurex Mountain View, California laboratories
- Preparing laboratory matrix spike solutions with the trace metals and analyzing these for spike recovery
- Preparing four hexavalent chromium field matrix spike samples at the IRF and analyzing these for spike recovery

TABLE 43. PRECISION, ACCURACY, AND COMPLETENESS OBJECTIVES

Measurement parameter	Measurement/ analytical method	Reference	Conditions	Precision (% RSD* or RPD**)	Accuracy (%)	Completeness (%)
Volatile organic compounds in clay/ liquid feed	Packed column GC/FID by the method in Appendix D	Appendix D	Direct injection	50	50-140	80
Volatile organic compounds on Tenax	Thermal desorption purge and trap by Method 5040, GC/FID analysis by the method in Appendix D	SW-846-3[a,b] Appendix D	Thermal desorption	100	50-150[b,c]	80
Cr(+6)	Methods 3060[d] and 7197[a]	SW-846-3[a], SW-846-2[d]	Alkaline digestion and AA analysis	25	75-12	80
Metals	Method 6010, 7000 series,[a] or 300 series[e] methods	SW-846-3[a], AWPCF[e]	Acid digestion and ICAP or AA analysis	25	75-125[f]	80

[a] (1)
[b] Recovery of surrogate spikes of resin samples greater than 20 percent was also an accuracy objective.
[c] Percent recovery from a spiked Tenax cartridge.
[d] (2)
[e] (7)
[f] Recovery of a spiked water solution.

*Relative Standard Deviation.
**Relative Percent Difference.

Results of these QA procedures are discussed in the following sections.

6.1 ORGANIC ANALYSIS OF CLAY/ORGANIC LIQUID FEED SAMPLES

As discussed in Section 3, a composite sample of the feed mixture from each drum prepared was collected and analyzed for the volatile organic hazardous constituents toluene, chlorobenzene, and tetrachloroethylene. Each drum of a given test mixture was prepared by mixing together weighed amounts of the individual liquid organic constituents, then mixing together weighed amounts of the liquid mixture and the clay sorbent. Waste feed for a given test was taken from one, two, or three drums depending on test conditions and durations. Thus, comparing analysis results for each drum of a target test mixture preparation gives a measure of the precision and accuracy of the entire feed preparation, sampling, and analysis procedure.

Table 44 provides such a comparison. For each target test mixture preparation, analysis results for a sample of each drum prepared are compared to the target composition and to each other. The accuracy of the feed preparation, sampling, and analysis operation is measured by the ratio of the analyzed concentration of a mixture component to the target value (termed recovery in Table 44). The precision of the procedure is measured by the relative standard deviation (RSD) of all samples analyzed.

The data in Table 44 show that analyzed concentrations ranged from 26 to 135 percent of target concentrations over 24 samples analyzed. Only 1 measurement out of 63 was out of the 50 to 140 percent recovery range, the accuracy DQO for the measurement. Thus, 98 percent of the measurements were acceptable. This DQO was met. The precision of the procedure as measured by the RSD of the analysis of different drum samples of a given target formulation ranged from 5 to 26 percent. All were within the precision DQO of 50 percent for this measurement. Thus, this DQO was met.

As a further measure of analytical precision, two clay/organic liquid feed samples were subjected to duplicate analysis. The relative percent difference (RPD) for these ranged from 1.3 to 10.1 percent for each of the constituents analyzed, as shown in Table 45. The precision DQO for the volatile organic compounds in the feed mixture was 50 percent RPD. This DQO was therefore met.

Three samples of a clay/organic liquid feed mixture matrix spike were prepared under controlled laboratory conditions (as opposed to bulk feed sample preparation) and analyzed to determine volatile organic constituent matrix spike recovery. The data in Table 46 show that the recoveries ranged from 75 to 104 percent. All were within the accuracy DQO range for this measurement of 50 to 140 percent recovery. Thus, this DQO was also achieved. Further, the RSD of the replicate spike analyses ranged from 8.9 to 11.3 percent, all within the DQO for precision of 50 percent.

It bears noting that the analytical data in Tables 44 and 45 are different from the feed mixture compositions used for DRE determinations in Section 4 (i.e., in Table 17). The data in Table 17 represent analyzed compositions (Tables 44 and 45) corrected for the matrix spike recoveries experienced (Table 46).

6.2 VOLATILE ORGANIC COMPOUNDS IN THE VOST TRAPS

To assess measurement accuracy of the VOST samples analyses, each resin trap was spiked with the surrogates octane and 4-bromofluorobenzene prior to use in sampling. Surrogate recovery was then measured.

TABLE 44. CLAY/ORGANIC LIQUID FEED SAMPLE POHC ANALYSIS RESULTS

Test	Test date	Drum	Toluene		Tetrachloroethylene		Chlorobenzene	
			Measured concentration (wt %)	Recovery[a] (%)	Measured concentration (wt %)	Recovery[a] (%)	Measured concentration (wt %)	Recovery[a] (%)
Mixture 1								
Target concentration			28.6		0		0	
1,2	9/28/88-9/29/88	1	26.4	92	—	—	—	—
		2	25.5	89	—	—	—	—
		3	24.4	85	—	—	—	—
4	9/14/88	1	22.5	79	—	—	—	—
		2	15.3	53	—	—	—	—
		3	26.6	93	—	—	—	—
RSD (%)				18				
Mixture 2								
Target concentration			21.7		3.4		3.4	
5	8/25/88	1	15.1	70	2.7	79	3.2	94
		2	15.7	72	2.3	68	3.0	88
6	9/16/88	1	19.1	88	3.1	91	3.0	88
		2	19.0	88	2.9	85	2.9	85
		3	18.9	87	3.1	91	3.2	94
7	8/30/88	1	16.0	74	2.3	68	2.5	74
		2	19.2	88	2.9	85	3.0	88
		3	19.4	89	2.7	79	3.0	88
8	9/7/88	1	16.3	75	2.8	82	2.7	79
		2	15.4	71	2.3	68	2.5	74
9	9/9/88	1	15.2	70	2.4	71	2.5	74
10	9/20/88	1	21.1	97	3.2	94	3.6	106
		2	20.1	93	0.9	26	4.6	135
		3	21.2	98	3.2	94	3.1	91
RSD (%)				15		26		18
Mixture 3								
Target concentration			14.9		6.9		6.9	
3	9/26/88	1	13.8	93	6.0	87	5.4	78
		2	15.3	103	6.4	93	6.0	87
11	9/22/88	1	12.8	86	5.6	81	5.7	83
		2	14.0	94	6.1	88	6.2	90
		3	13.6	91	6.0	87	6.0	87
RSD (%)				7		5		5

[a] Ratio of measured concentration to target concentration.

TABLE 45. CLAY/ORGANIC LIQUID FEED REPLICATE VOLATILE ORGANIC ANALYSIS RESULTS

			Toluene			Tetrachloroethylene			Chlorobenzene		
Test	Test date	Drum	1st Analysis (wt %)	2nd Analysis (wt %)	RPD (%)	1st Analysis (wt %)	2nd Analysis (wt %)	RPD (%)	1st Analysis (wt %)	2nd Analysis (wt %)	RPD (%)
3	9/26/88	2	15.3	15.1	1.3	6.4	6.3	1.4	6.0	5.9	1.9
1,2	9/28/88- 9/29/88	3	24.4	27.0	10.1						

TABLE 46. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM CLAY/ORGANIC LIQUID FEED MATRIX SPIKE SAMPLES

	Percent recovery			
Compound	Spike	Replicate spike	Replicate spike	Relative Standard Deviation (RSD)
Toluene	86	88	104	8.9
Chlorobenzene	78	81	100	11.3
Tetrachloroethylene	75	79	95	10.4

Table 47 summarizes octane recovery from the VOST trap pairs. The data in the table show that octane recovery ranged from 0 to 217 percent, with 7 of 75 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 91 percent, and met the DQO for recovery completeness of 80 percent.

Table 48 similarly summarizes 4-bromofluorobenzene recovery from the VOST trap pairs. The data show that 4-bromofluorobenzene recovery ranged from 0 to 120 percent, with 3 of 75 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 96 percent, and met the DQO for recovery completeness of 80 percent.

As a further measure of VOST analysis accuracy, and as a measure of analysis precision, seven matrix spike VOST trap pairs were prepared and analyzed at the IRF. Results of these analyses are summarized in Table 49. The data in the table show that matrix spike recoveries ranged from 73 to 194 percent, with 2 of 35 recoveries not within the DQO range of 50 to 150 percent. Measurement completeness was 94 percent, and met the DQO for recovery completeness of 80 percent. Further, the RSD of the matrix spike analyses ranged from 3.6 to 29 percent, all within the DQO for precision of 100 percent.

In addition to the seven traps analyzed at the IRF, an eighth matrix spike VOST trap pair was prepared and shipped to Acurex's Mountain View, California, laboratory for analysis by EPA Method 5040. This spike sample was prepared identically to one analyzed at the IRF. Table 50 summarizes the analysis results for this VOST trap pair and compares results of the Method 5040 GC/MS analysis with the GC/FID analysis of an identically prepared trap pair. Matrix spike and surrogate recoveries for the GC/MS-analyzed sample ranged from 46 to 137 percent, with one out of eight compounds not within the DQO recovery range of 50 to 150 percent. Measurement completeness was 87 percent, and met the DQO for recovery completeness of 80 percent. In addition, the RPD between the GC/MS-analyzed traps and the GC/FID-analyzed traps ranged from 28 to 80 percent, all within the precision DQO for this measurement of 100 percent RPD.

To further compare VOST trap analysis results between the GC/FID analysis method routinely performed at the IRF (see Appendix D) and the GC/MS Method 5040 analysis method, two sampling trains were operated simultaneously at the scrubber exit during Test 10 to obtain two sets of VOST traps from the same flue gas. One set was analyzed at the IRF by GC/FID (Appendix D), and one set was analyzed by Method 5040 at the Acurex Mountain View laboratory. Unfortunately, the GC/MS analysis of one of the trap pairs (the third pair) was invalidated due to a leak during analysis. Table 51 summarizes the VOST results reported for each method.

The precision DQO for the measurement of volatile organic compounds in VOST traps is 100 percent RPD. The data in Table 51 show that reasonably good agreement between the two analysis methods, within about 100 percent RPD, was achieved for chloroform, bromodichloromethane, dibromochloromethane, bromoform, toluene, chlorobenzene, and tetrachloroethylene (the last three compounds being the POHCs for these tests). However, agreement between the two analysis methods was not as good for carbon tetrachloride, benzene, dichlorobenzenes, and 1,2-dichloroethane. Surrogate recoveries were acceptable by both methods. Given the mixed nature of these results, further method comparisons are recommended and planned in future tests.

TABLE 47. OCTANE SURROGATE RECOVERY FROM VOST TRAPS

Afterburner exit traps		Scrubber system exit traps		Other traps	
Sample	Octane recovery (%)	Sample	Octane recovery (%)	Sample	Octane recovery (%)
				Stack Samples	
Test 4 (9/14/88)		Test 4 (9/14/88)		Test 8 (9/07/88)	
1st trap pair	121	1st trap pair	118	1st trap pair	123
2nd trap pair	101	2nd trap pair	107	2nd trap pair	109
3rd trap pair	76	3rd trap pair	109	3rd trap pair	134
Test 5 (8/25/88)		Test 5 (8/25/88)		Test 9 (9/09/88)	
1st trap pair	137	1st trap pair	217	1st trap pair	102
2nd trap pair	151	2nd trap pair	112	2nd trap pair	34
3rd trap pair	117	3rd trap pair	153	3rd trap pair	119
Test 6 (9/16/88)		Test 6 (9/16/88)		Test 11 (9/22/88)	
1st trap pair	107	1st trap pair	109	1st trap pair	91
2nd trap pair	114	2nd trap pair	111	2nd trap pair	122
3rd trap pair	115	3rd trap pair	0	3rd trap pair	123
Test 7 (8/30/88)		Test 7 (8/30/88)		**Field blank traps**	
1st trap pair	114	1st trap pair	111		
2nd trap pair	110	2nd trap pair	111	8/25/88-1	36
3rd trap pair	76	3rd trap pair	98	8/25/88-2	94
				8/30/88	80
Test 8 (9/07/88)		Test 8 (9/07/88)		9/07/88	80
1st trap pair	117	1st trap pair	96	9/09/88	120
2nd trap pair	136	2nd trap pair	100	9/14/88	101
3rd trap pair	108	3rd trap pair	114	9/16/88	105
				9/20/88	106
Test 9 (9/09/88)		Test 9 (9/09/88)		9/22/88	100
1st trap pair	79	1st trap pair	133		
2nd trap pair	116	2nd trap pair	135	**Laboratory method blank traps**	
3rd trap pair	116	3rd trap pair	118	8/25/88	72
Test 10 (9/20/88)		Test 10 (9/20/88)			
1st trap pair	122	1st trap pair	81	**Matrix spike traps**	
2nd trap pair	117	2nd trap pair	117	8/25/88	98
3rd trap pair	115	3rd trap pair	178	8/30/88	103
				9/07/88	108
Test 11 (9/22/88)		Test 11 (9/22/88)		9/09/88	104
1st trap pair	115	1st trap pair	111	9/14/88	107
2nd trap pair	0	2nd trap pair	117	9/16/88	109
3rd trap pair	111	3rd trap pair	112	9/20/88	112
				9/22/88	105

TABLE 48. 4-BROMOFLUOROBENZENE SURROGATE RECOVERY FROM VOST TRAPS

Afterburner exit traps		Scrubber system exit traps		Other traps	
Sample	**4-Bromo-fluoro-benzene recovery (%)**	**Sample**	**4-Bromo-fluoro-benzene recovery (%)**	**Sample**	**4-Bromo fluoro-benzene recovery (%)**
				Stack Samples	
Test 4 (9/14/88)		Test 4 (9/14/88)		Test 8 (9/07/88)	
1st trap pair	107	1st trap pair	100	1st trap pair	96
2nd trap pair	86	2nd trap pair	104	2nd trap pair	98
3rd trap pair	81	3rd trap pair	98	3rd trap pair	111
Test 5 (8/25/88)		Test 5 (8/25/88)		Test 9 (9/09/88)	
1st trap pair	105	1st trap pair	91	1st trap pair	120
2nd trap pair	115	2nd trap pair	105	2nd trap pair	108
3rd trap pair	101	3rd trap pair	112	3rd trap pair	120
Test 6 (9/16/88)		Test 6 (9/16/88)		Test 11 (9/22/88)	
1st trap pair	83	1st trap pair	104	1st trap pair	79
2nd trap pair	101	2nd trap pair	106	2nd trap pair	108
3rd trap pair	105	3rd trap pair	0	3rd trap pair	107
Test 7 (8/30/88)		Test 7 (8/30/88)		**Field blank traps**	
1st trap pair	94	1st trap pair	99		
2nd trap pair	99	2nd trap pair	95	8/25/88-1	27
3rd trap pair	80	3rd trap pair	37	8/25/88-2	94
				8/30/88	81
Test 8 (9/07/88)		Test 8 (9/07/88)		9/07/88	88
1st trap pair	104	1st trap pair	104	9/09/88	96
2nd trap pair	115	2nd trap pair	81	9/14/88	97
3rd trap pair	95	3rd trap pair	103	9/16/88	102
				9/20/88	94
Test 9 (9/09/88)		Test 9 (9/09/88)		9/22/88	88
1st trap pair	89	1st trap pair	102		
2nd trap pair	92	2nd trap pair	101	**Laboratory method blank traps**	
3rd trap pair	106	3rd trap pair	102		
				8/25/88	92
Test 10 (9/20/88)		Test 10 (9/20/88)			
1st trap pair	107	1st trap pair	101	**Matrix spike traps**	
2nd trap pair	104	2nd trap pair	91	8/25/88	97
3rd trap pair	92	3rd trap pair	110	8/30/88	96
				9/07/88	93
Test 11 (9/22/88)		Test 11 (9/22/88)		9/09/88	110
1st trap pair	96	1st trap pair	106	9/14/88	105
2nd trap pair	110	2nd trap pair	97	9/16/88	106
3rd trap pair	105	3rd trap pair	105	9/20/88	105
				9/22/88	98

TABLE 49. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM VOST MATRIX SPIKE SAMPLES

Compound	Percent recovery							Relative Standard Deviation (% RSD)
	Test 4 (9/14/88)	Test 5 (8/25/88)	Test 7 (8/30/88)	Test 8 (9/07/88)	Test 9 (9/09/88)	Test 10 (9/20/88)	Test 11 (9/22/88)	
1,1,1-Trichloroethane	111	93	107	113	98	103	126	9.4
Benzene	73	116	130	127	194	92	153	29.0
Toluene	123	124	115	147	124	131	121	10.0
Tetrachloroethylene	104	103	101	100	107	111	100	3.6
Chlorobenzene	102	96	100	100	108	107	97	4.2

TABLE 50. VOLATILE ORGANIC CONSTITUENT RECOVERY FROM THE VOST MATRIX SPIKE SAMPLE FOR TEST 10 (9/20/88) BY METHOD 5040 AND GC/FID ANALYSIS

Compound	Percent recovery		RPD (%)
	GC/FID	GC/MS	
1,1,1-Trichloroethane	103	64	47
Benzene	92	65	34
Toluene	131	71	59
Tetrachloroethylene	111	84	28
Chlorobenzene	107	46	80
1,1-Dichloroethane	—[a]	137	—
4-Bromofluorobenzene[b]	105	57	59
Toluene-d8[b]	—[a]	87	—

[a] Not reported.
[b] Method surrogates.

TABLE 51. VOLATILE ORGANIC CONSTITUENT CONCENTRATIONS FOR DUPLICATE VOST SAMPLES TAKEN AT THE SCRUBBER EXIT DURING TEST 10 (9/20/88) AND ANALYZED BY GC/FID AND GC/MS (METHOD 5040)

	Flue gas concentration (µg/dscm)					
	1st Trap pair		Relative Percent Difference	2nd Trap pair		Relative Percent Difference
Compound	**GC/FID**	**GC/MS**		**GC/FID**	**GC/MS**	
Chloroform	22.0	14.7	40	12.2	2.7	128
Carbon Tetrachloride	3.9	0.7	139	1.5	<0.25	—
Benzene	19.2	0.7	186	8.4	<0.25	—
Bromodichloromethane	12.7	16.9	28	3.8	4.8	23
Dibromochloromethane	27.3	11.6	81	77.6	7.0	167
Bromoform	11.6	10.2	13	15.7	15.0	4.6
1,2-Dichloroethane	3.2	<0.2	—	2.2	<0.25	—
1,4-Dichlorobenzene	4.4	<0.2	—	<1.5	<0.25	0
1,2-Dichlorobenzene	6.5	<0.2	—	4.6	<0.25	—
Toluene	19.1	13.4	35	15.5	10.0	43
Chlorobenzene	1.7	1.1	43	<1.6	0.9	0
Tetrachloroethylene	2.7	2.5	7.7	4.8	1.6	102
Surrogate Recoveries (%)						
Octane	117	—[a]	—	81	—[a]	—
4-bromofluorobenzene	91	65	33	101	79	24
Toluene-d8[b]	—	99	—	—	80	—

[a] GC/FID surrogate only.
[b] GC/MS surrogate only.

6.3 METALS ANALYSIS

For metals analysis in the laboratory, matrix spike and matrix spike duplicate samples were prepared in the laboratory and analyzed to assess analytical precision and accuracy. In addition, field matrix spike and spike duplicate samples were prepared at the IRF and shipped to the Mountain View laboratory for analysis for the chromium valence state tests. Analysis results are discussed in the following subsections.

6.3.1 Chromium Valence State Tests

Table 52 summarizes the results of the laboratory matrix spike/matrix spike duplicate analyses for the chromium valence state test series. Indicated in the table are the group of samples to which the matrix spike samples apply. Matrix spike recovery for laboratory-prepared total chromium and Cr(+6) samples ranged from 89 to 115 percent, all within the DQO range of 75 to 125 percent. The RPD between the values obtained for the matrix spikes and the matrix spike duplicates ranged from 0 to 24 percent. All values were within the DQO for measurement precision of 25 percent.

Table 53 summarizes the results of the Cr(+6) spike analyses of field matrix spike samples prepared at the IRF. Two sets of spike recovery values are noted in the table, one set obtained from analyses performed 12 days after sample preparation, and one set obtained 69 days after sample preparation. The QAPP for these tests specified an analytical hold time for Cr(+6) samples of 7 days between sample collection and analysis. However, between 11 and 12 days actually elapsed between collection and analysis. As a consequence, it was decided to reanalyze the matrix spike samples after a second period of hold time to evaluate the possible effects of exceeding hold time on test conclusions.

The data in Table 53 show that Cr(+6) matrix spike recovery ranged from 32 percent for the feed clay matrix to 90 percent for the kiln ash matrix for the original analysis. A range of recoveries is noted for the scrubber blowdown matrix. This is because a blank sample of the actual matrix spiked was not analyzed. Instead, blank samples with background Cr(+6) concentrations which were known to bracket the actual matrix spiked were analyzed. Thus, actual matrix spike recovery would been in the range noted in Table 53.

The accuracy DQO for this measurement was 75 to 125 percent recovery. Kiln ash, aqueous feed spike solution, and possibly scrubber blowdown matrix spike recovery for the original analyses met this objective. Cr(+6) recoveries from the feed clay spike recoveries were low. Furthermore, the precision of the measurement was 48 percent RPD, greater than the 25 percent DQO for the measurement.

Matrix spike recoveries were uniformly increased for the second analyses series, performed 8 weeks after the original analyses. For these analyses spike recoveries were within the DQO range for all samples except the scrubber blowdown. The precision of the feed clay matrix spike duplicate analyses, at 14 percent RSD, was also within the DQO.

The fact that better QA measurement parameter performance was achieved after samples had aged considerably suggests that exceeding sample hold time did not affect the quality of the data reported. With respect to the inability to uniformly achieve the DQOs in the original sample analyses, it must be noted that the sample matrix failing the spike recovery and precision objective was the feed clay matrix. Acceptable spike recovery was achieved for the matrices of more importance in the tests—the matrices whose analyses were used to formulate

TABLE 52. SUMMARY OF LABORATORY MATRIX SPIKE SAMPLE ANALYSIS RESULTS FOR THE CHROMIUM VALENCE STATE TEST SERIES

Test	Analyte	Corresponding sample	Spike recovery (%)	Duplicate spike Relative Percent Difference (%)
1, 2, 3	Total Cr	Ash	115	7.9
1	Total Cr	Water[a]	105	1.9
2	Total Cr	Water[a]	101	0.2
3	Total Cr	Water[a]	98	1.6
1, 2, 3	Total Cr	Afterburner exit train filter	89	0.9
1	Total Cr	Scrubber exit train filter	89	0.9
2	Total Cr	Scrubber exit train filter	89	0.9
3	Total Cr	Scrubber exit train filter	89	0.9
1	Cr(+6)	All	94	24
2	Cr(+6)	All	94	6.2
3	Cr(+6)	All	102	0

[a] Applies to feed, impinger, and probe wash samples.

TABLE 53. CR(+6) MATRIX SPIKE RECOVERIES

Matrix	Cr(+6) Concentration (ppm)				Spike recovery (%)	
	Blank level	Spiked level	Analyzed			
			Original[a]	Repeat[b]	Original[a]	Repeat[b]
Feed clay	2.3	10	3.9	10.8	32	88
Feed clay duplicate	2.3	10	6.4	12.4	52	101
RPD (percent)					48	14
Scrubber blowdown[c]	0.10 to 0.28	0.10	0.19	0.49	50 to 95	129 to 245
Kiln ash	0.27	10	9.2	10.8	90	105
Aqueous feed spike	—	3,400	2,700	3,100	79	91

[a] Analyzed 12 days after sample preparation.
[b] Analyzed 69 days after sample preparation (8 weeks after original analysis).
[c] Matrix blank samples which bracket the matrix spike sample were analyzed.

test conclusions. These were the kiln ash, aqueous feed spike solution, and possibly the scrubber blowdown. Given this, it is felt that test conclusions are valid as stated in Section 5.

6.3.2 Parametric Trace Metal Tests

Table 54 summarizes the trace metal recoveries for the laboratory matrix spikes corresponding to analysis of water-related samples for the parametric test series. These spike and duplicate spike samples apply to aqueous feed spike solutions, impinger, blowdown and probe wash samples obtained during the trace metals parametric test series. Matrix spike recovery for the trace metal constituents ranged from 81 to 123 percent, all within the DQO for recovery of 75 to 125 percent. The RPD between the values obtained for the matrix spike and the matrix spike duplicates ranged from 0 to 37 percent. One out of 72 duplicate spike pairs did not fall within the DQO limit for precision of 25 percent. Measurement completeness was 98 percent and met the DQO for precision completeness of 80 percent.

Table 55 summarizes the trace metal recoveries for the laboratory matrix spikes corresponding to analysis of filter and ash samples for the parametric test series. For the kiln ash samples, matrix spike recovery of the trace metal constituents ranged from 72 to 137 percent. Five of the 27 spike samples were not within the DQO range for recovery of 75 to 125 percent. Measurement completeness was 81 percent, and met the DQO for recovery completeness of 80 percent. The RPD between the values obtained for the matrix spike and matrix spike duplicates ranged from 0.3 to 27 percent. One out of 27 duplicate spikes exceeded the DQO for precision

TABLE 54. LABORATORY MATRIX SPIKE AQUEOUS SAMPLE ANALYSIS RESULTS FOR THE PARAMETRIC TEST SERIES

	Test 4 (9/14/88)		Test 5 (8/25/88)		Test 6 (9/16/88)		Test 7 (8/30/88)		Test 8 (9/07/88)		Test 9 (9/09/88)		Test 10 (9/20/88)		Test 11 (9/22/88)	
Metal	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)
Arsenic	86	9.2	86	5.9	88	3.4	95	3.3	81	1.6	86	4.2	89	9.3	94	5.3
Barium	97	7.6	97	0.6	105	2.7	101	3.6	105	3.0	104	0.9	93	4.2	96	3.9
Bismuth	123	37	88	17	98	1.0	93	14	90	15	83	0.8	84	7.4	114	13
Cadmium	103	5.2	101	0.3	106	2.5	106	2.8	105	0.9	102	1.7	101	2.9	98	4.3
Chromium	97	6.5	97	0.2	108	2.6	102	2.8	104	2.2	102	0.8	97	3.9	98	2.9
Copper	92	7.6	92	0.5	102	2.4	96	3.6	99	2.3	84	2.0	83	2.9	95	4.0
Lead	105	11	104	1.9	116	6.4	104	11	106	6.7	122	2.1	109	5.1	100	3.4
Magnesium	94	5.3	91	0.5	103	1.0	95	4.5	107	2.4	98	1.4	91	2.8	97	2.2
Strontium	108	5.1	101	0	106	3.1	105	2.9	109	0.9	103	3.3	107	2.6	117	6.3

TABLE 55. LABORATORY MATRIX SPIKE FILTER AND ASH SAMPLE ANALYSIS RESULTS FOR THE PARAMETRIC TEST SERIES

	Kiln Ash						Afterburner exit sampling train filter		Scrubber exit sampling train filter			
	Tests 4,5,8,9,10,11		Test 6		Test 7		Tests 4,6,7,8,9,11		Tests 4,7,9		Tests 6,10,11	
Metal	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)	Spike recovery (%)	Duplicate spike RPD (%)
Arsenic	125	6.5	117	91	124	18	105	5.6	77	19	70	3.6
Barium	106	1.3	80	2.1	111	6.9	100	3.7	104	3.7	90	1.2
Bismuth	136	13	101	27	91	3.6	92	22	64	16	93	1.1
Cadmium	86	1.7	93	0.3	98	9.1	110	3.9	93	0.4	91	2.5
Chromium	93	5	90	3.1	115	11	105	6.0	94	1.0	89	0.9
Copper	137	5.7	72	1.3	91	18	92	4.1	94	2.2	77	1.9
Lead	111	18	104	1.6	117	11	108	3.5	100	0	96	1.7
Magnesium	81	5.1	84	7.3	100	3.4	81	9.7	122	13	102	4.3
Strontium	129	7.6	72	4.3	104	20	97	3.0	104	23	123	14

of 25 percent. Measurement completeness was 96 percent and met the DQO for precision completeness of 80 percent.

For the filter samples, matrix spike recovery of the trace metal constituents ranged from 64 to 123 percent. Two of 27 spike samples were not within the DQO range for recovery of 75 to 125 percent. Measurement completeness was 92 percent, and met the DQO for recovery completeness of 80 percent. The RPD between the values obtained for the matrix spike and matrix spike duplicates ranged from 0 to 23 percent, all within the DQO for precision of 25 percent.

As a further measure of trace metal analytical accuracy and precision, recall from Section 4.1 that a composite sample of the aqueous metal spike solution for each test was analyzed for the 9 test trace metals. These can be considered field aqueous solution matrix spike samples. Comparing analysis results from test to test will give a measure of the accuracy and precision of the entire aqueous spike solution preparation, sample collection and preservation, and laboratory analysis process.

Table 19 summarized the results of all aqueous metal spike solution analyses. Table 56 presents these results in different form. Specifically, Table 56 tabulates aqueous spike solution matrix spike recoveries where recovery in defined as the analyzed concentration divided by the target preparation concentration. The data in Table 56 show that analysis recoveries ranged from 67 to 122 percent. Seven of 8 analyses were within the recovery DQO for this measurement of 75 to 125 percent for all the metals except arsenic. Thus, measurement completeness was 88 percent for all metals except arsenic, compared to a completeness DQO of 80 percent. Only 4 of 8 arsenic spike recoveries were between 75 and 125 percent, for a completeness of 50 percent. Arsenic measurement accuracy failed its DQO on this basis. However, the failing recoveries were between 71 and 74 percent. These marginally low recoveries suggest that failure to achieve the recovery DQO for arsenic from this matrix would have no impact on test program conclusions regarding arsenic distributions.

The data in Table 56 also show that the precision of individual metal analyses for the aqueous spike solutions ranged from 9 to 18 percent RSD. All precisions were within the DQO for this measurement of 25 percent. Thus, the measurement precision DQO was met.

Finally, recall from Section 4.1 that clay matrix samples for all parametric trace metal tests were analyzed for lead, magnesium, and strontium, and for all tests were analyzed for chromium. Comparing all these analyses gives a measure of the analytical precision of these metal analyses in the clay matrix. Table 57 provides this comparison. As indicated in Table 57, the demonstrated analytical precision for the four metals ranged from 3.6 to 16 percent RSD. Again, all these results were within the precision DQO of 25 percent RSD.

TABLE 56. AQUEOUS SPIKE SOLUTION ANALYSIS ACCURACY AND PRECISION

Metal	Prepared spike solution concentration (ppm as metal)	Analysis recovery (%)[a]								
		Test 4 (9/14/88)	Test 5 (8/25/88)	Test 6 (9/16/88)	Test 7 (8/30/88)	Test 8 (9/07/88)	Test 9 (9/09/88)	Test 10 (9/20/88)	Test 11 (9/22/88)	RSD (%)
Arsenic	3230	87	71	90	74	74	71	111	102	18
Barium	2580	89	70	93	93	97	97	97	89	10
Bismuth	9040	111	73	122	82	104	122	122	122	18
Cadmium	650	93	63	83	82	80	83	98	83	12
Chromium	3230	96	71	90	90	96	93	102	90	10
Copper	32,300	90	71	93	93	99	96	93	93	9
Lead	3230	105	74	93	96	99	96	108	93	11
Magnesium	5040	91	67	89	85	87	91	97	85	10
Strontium	16,100	112	68	93	87	93	99	118	106	16

[a]Analyzed amount (see Table 19)/prepared amount.

TABLE 57. FEED CLAY ANALYSIS PRECISION

	Clay sample metal concentration (ppm)										
Metal	Test 1/2 composite (9/28,29/88)	Test 3 (9/26/88)	Test 4 (9/14/88)	Test 5 (8/25/88)	Test 6 (9/16/88)	Test 7 (8/30/88)	Test 8 (9/07/88)	Test 9 (9/09/88)	Test 10 (9/20/88)	Test 11 (9/22/88)	RSD (%)
Chromium	54	52	55	55	54	53	54	55	50	50	3.6
Lead	NA[a]	NA	3.2	3.7	3.5	2.9	3.1	2.3	3.0	2.5	16
Magnesium	NA	NA	23,000	23,000	22,000	22,000	22,000	22,000	20,000	22,000	4.2
Strontium	NA	NA	33	37	36	32	33	30	33	35	6.7

[a] NA: not analyzed.

REFERENCES

1. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. EPA SW-846, 3rd ed., U.S. Environmental Protection Agency, Washington, D.C., November 1986.

2. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. EPA SW-846, 2nd ed., U.S. Environmental Protection Agency, Washington, D.C., July 1982.

3. Harris, J. C., et al. Sampling and Analysis Methods for Hazardous Waste Incineration. EPA-600/8-84-002, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, February 1984.

4. Lentzen, D. E., et al. IERL-RTP Procedures Manual: Level 1 Environmental Assessment (Second Edition). EPA-600/7-78-201, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina, October 1978.

5. 40 CFR Part 60, Appendix A.

6. Determining the Properties of Fine Particulate Matter. ASME Power Test Code 28.

7. Standard Methods for the Examination of Water and Wastewater. 16th Edition, APHA, AWWA, WPCF, 1985.

8. Barton, R. G., et al. Development and Validation of a Surrogate Metals Mixture. In: Proceedings of the Fifteenth Annual Research Symposium: Remedial Action, Treatment and Disposal of Hazardous Waste. U.S. Environmental Protection Agency, Cincinnati, Ohio, April 1989.